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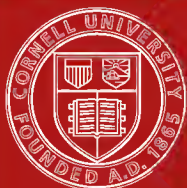
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ENGINEERING CHEMISTRY

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ENGINEERING CHEMISTRY

A PRACTICAL TREATISE

FOR THE USE OF

Analytical Chemists, Engineers, Ironmasters, Ironfounders
Students, and Others

COMPRISING

METHODS OF ANALYSIS AND VALUATION OF THE PRINCIPAL MATERIALS USED IN ENGINEERING WORK
WITH NUMEROUS ANALYSES, EXAMPLES
AND SUGGESTIONS

BY

H. JOSHUA PHILLIPS, F.I.C., F.C.S.

ANALYTICAL AND CONSULTING CHEMIST TO THE GREAT EASTERN RAILWAY

AUTHOR OF

"FUELS—SOLID, LIQUID, AND GASEOUS: THEIR ANALYSIS AND VALUATION"



LONDON

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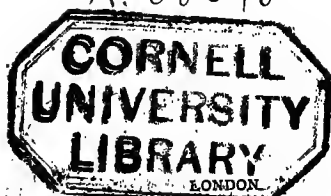
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PREFACE.

IN the Author's capacity as an analytical chemist, engaged in the laboratories of railway companies, he has frequently heard it remarked by pupils, whether in chemistry or in engineering, as well as by practising engineers and others, that a work which should give precise methods for analysing and valuing the most important of the materials in general use by engineers, would be of great service. Having himself also felt the want of such a book when a student, he has utilised a portion of his leisure time in collecting the materials for the present work, which he trusts will meet with general approbation, and be found to sufficiently answer the purpose for which it has been written.

It will be observed that Reddrop's "equivalent system" of reagents, of which the Author has now had seven years' experience, is used as far as possible throughout the work. This system he considers pre-eminently the best for adoption in analytical chemistry, insuring, as it does, accuracy and uniformity of results, and limiting the "personal equation" to a great extent. He is indebted to the courtesy of Mr. W. Crookes, F.R.S., and of Mr. Reddrop himself, for the full description of the system as given by its

author in the *Chemical News* of May 23rd and 30th, 1890.

Many of the methods of analysis given in the following pages, as far as the strength and volumes of solutions are concerned, were worked out by the writer when acting as Chief Assistant in the Laboratory of the Great Western Railway, under the direction of Mr. F. W. Harris, F.I.C., to whom he desires to acknowledge his indebtedness for much of his knowledge of chemical analysis. He has also to express his obligations to the authors of the various published works, papers in the Proceedings of Societies, etc., which have been consulted by him, and to which in many cases direct reference is made in the body of the work.

The substance of a smaller work* which the Author had already published, and which has been very cordially received by chemists and engineers, has been embodied, with some slight additions, in this volume (Part III.).

In conclusion, he would say that if the present work prove acceptable, he will gladly avail himself in future editions of any suggestions with which he may be favoured by any of his readers, or for which he may be indebted to notices of the book in the public press.

STRATFORD, LONDON, E.,

July, 1891.

* "Fuels—Solid, Liquid, and Gaseous : Their Analysis and Valuation."
London : Crosby Lockwood & Son.

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ENGINEERING CHEMISTRY.

INTRODUCTORY CHAPTER.

REDDROP'S SYSTEM OF REAGENTS.

I THINK it will be generally agreed that many of the text books and standard works on Analytical Chemistry rely rather too much on the discretion of the operator as to the strength and volume of the solutions, &c., which are to be employed in the work of analysis; and if the analyst attempts to follow new methods as he finds them described in scientific periodicals, it is often very difficult to obtain such satisfactory results as the authors of such methods claim for them, owing to the want of a precise and universal system of detailing the *modus operandi* of the processes.

The discordant results of analysis, often obtained by different chemists operating upon the same sample, are much to be regretted; and as the discrepancies are mainly due to differences of working, it is to be hoped that something will soon be done to systematize methods of analysis generally, so that there may be no excuse for differences of result.

A practical insight into chemistry, as affecting engineering, is now considered essential for the complete education of an engineer. As a pupil, the young engineer serves from three to six months in the works' laboratory, and a precise description of the methods of analysis of materials coming under the cognisance of the practising engineer should prove of great service to an engineering pupil, as well as a means of relief

to his tutor, who is often preoccupied otherwise than in teaching.

The two main points to be considered in describing a method of analysis are, (1) The *strength* of reagents used ; and (2) The *volume* of reagents employed. It therefore becomes necessary to have a stock of reagents of definite strength at hand in the laboratory. As a general rule, in instructions for analysis, the specific gravity of the desired solution or one part of a strong reagent diluted so many times, is referred to as a means of indicating the solution to be employed; but this mode of expressing strength is not very definite or convenient. The best system of reagents with which I am acquainted is that devised by Mr. J. Reddrop, F.I.C., the chemist of the London and North-Western Railway. This system (it will be found) has been followed as far as possible throughout this work, my own experience having led me to fully endorse the claims of its author (see *post*, page 14) as to the advantages to be secured by the employment of the system.

In the year 1877 (Mr. Reddrop states) he introduced into the new laboratory at Crewe a number of reagents, the strength of which was based on the chemical equivalents. Their use was subsequently extended until it included almost all the reagents in the laboratory, and the advantages derived therefrom were found to be so decided, especially in chemical analysis, that they are now employed in all investigations at the Crewe laboratory, and also at the laboratories of the Great Western Railway at Swindon, and the Great Eastern Railway at Stratford.

Subjoined is a full description of the system as given by Mr. Reddrop * :—

Reddrop's System of Chemical Reagents Based on the Equivalents of the Elements.

An equivalent reagent may be defined as one which contains a milligram equivalent of reacting substance in one cc. of solution, or a grm. equivalent in one litre. A solution of this strength

* Vide *Chemical News*, May, 1890.

is called an equivalent solution, or an equivalent reagent, and is denoted by the symbol E, *e.g.*, E sodium carbonate represents a solution containing 53 m.grms., = 0.053 gm. in 1 cc., or 53 grms. in 1 litre. It will be observed that this is not a *percentage solution*, but one containing parts by *weight* (m.grms.) of substance, in parts by *volume* (cc.) of solution. The strengths of all the reagents are expressed in terms of E,* thus the strength of sulphuric acid, sp. gr. 1.84, is approximately 36 E; strong nitric acid, sp. gr. 1.42, is about 16 E; and strong hydrochloric acid, sp. gr. 1.16, is about 10 E. Many of the reagents employed are made of E strength, but in some cases the substance is not sufficiently soluble to make an E solution, and consequently some fractional part thereof has to be adopted instead.

Thus we have $\frac{E}{4}$ hydrogen sulphide, $\frac{E}{20}$ calcium oxide, $\frac{E}{30}$ calcium sulphate, $\frac{E}{600}$ strontium sulphate (test for barium). The fractional indices may, if preferred, be expressed decimally, thus $\frac{3E}{5}$ or 0.6 E. All E reagents are equal in precipitating or *quantitative* reacting power. For example, if we have a quantity of lead salt in solution which requires 36 cc. of E sulphuric acid to exactly precipitate the lead salt as sulphate (not allowing for free sulphuric acid in excess), it will require the same number of cc. of E ammonium carbonate to precipitate the lead as carbonate, or of E potassium chromate to precipitate the lead as chromate. The same quantity of lead in solution could, however, be precipitated by 1 cc. of 36 E sulphuric acid, or by 7.2 cc. of 5 E ammonium carbonate, but it would require at least 144 cc. of $\frac{E}{4}$ hydrogen sulphide (saturated solution) to precipitate the lead as sulphide.

It may here be pointed out that $\frac{E}{x}$ reagents are not made with the same degree of accuracy as the *Standard* E reagents employed in volumetric analysis. Variations in strength to the

* E being used as the symbol of equivalent or equivalents.

extent of from $\frac{1}{100}$ th to $\frac{1}{20}$ th part are not considered inadmissible; they may, consequently, be made in almost the same time as those hitherto employed, and the strength, in terms of E, is printed on the label of each bottle, thus—

SULPHURIC ACID.

36 E.

or

SULPHURIC ACID.

36 $\frac{\text{H}_2\text{SO}_4}{2}$.

or

SULPHURIC ACID.

36 E.

E. = $\frac{\text{H}_2\text{SO}_4}{2}$.

The accompanying Table includes the reagents to which the equivalent system has already been extended.

Column 1 gives the name of the reagent.

Column 2 the molecular formula of the anhydrous substance.

Column 3 the molecular weight of the anhydrous substance.

Column 4 the equivalent weight of the anhydrous substance.

Column 5 gives the strength of reagent as recommended by Fresenius, "Qual. Analysis," 10th English Edition, first in the terms stated by him, and second, as calculated on the equivalent system. This double column is simply given for comparison.

Column 6 gives the strength of reagent recommended on

the equivalent system, first in grms. per litre, and second in terms of E.

Column 7 gives the chemical formula of the substances used, and the methods by which the reagents are prepared of *sufficient accuracy for general use*.

Column 8 gives the strength of reagent as determined by trial after being prepared by the method given.

Several notes are also appended, to which the reader should refer for further information.

NOTES TO TABLE I.

(1) Sulphuric acid, sp. gr. 1·842, containing 98·6 per cent. by calculation gives 37·07 E.

Sulphuric acid, sp. gr. 1·84, as supplied, two samples titrated gave 36·6 E and 36·8 E.

(2) I usually make 5 litres of 5 E acid at a time and adjust the strength by titrating 2 cc. with standard N sodium hydrate. This can be done more quickly than by using the hydrometer, owing to changes of temperature on dilution. 5 E ammonium hydrate may be made in a similar manner. These 5 E reagents may also be made by simply diluting the concentrated ones to the right degree.

(3) Nitric acid, sp. gr. 1·50, containing nitrous acid, titrated, gave 22·8 E.

(4) Nitric acid, sp. gr. 1·424, containing 70·2 per cent., by calculation gives 15·87 E.

Nitric acid, sp. gr. 1·42, as supplied, two samples titrated gave 16·05 E and 16·6 E.

(5) Hydrochloric acid, sp. gr. 1·160, containing 31·73 per cent. by calculation, gives 10·08 E.

Hydrochloric acid, sp. gr. 1·16, as supplied, two samples titrated gave 10·36 E and 10·32 E.

(6) Carbonic acid. Water at 15° C. dissolves 1·0020 vols. of CO₂; this, by calculation, gives $\frac{100 \text{ E}}{1114}$.

Column 1.	2.	3.	4.	5. FRESSENIUS'S SYSTEM.	
Name of Reagent.	Symbol.	Mole- cular weight.	EQUIVA- lent weight.	Strength of Reagent. As stated.	Calculated in equivalent- weights.
Sulphuric Acid	H_2SO_4	98	49	Sp. gr. 1.840	36 E
"	"	"	"	1 to 5	6 E
Nitric Acid	HNO_3	63	63	—	—
"	"	"	"	—	—
"	"	"	"	Sp. gr. 1.2	6.16 E
Hydrochloric acid	HCl	36.5	36.5	—	—
"	"	"	"	Sp. gr. 1.12	7.48 E
"	"	"	"	—	—
Sulphurous acid	H_2SO_3	82	41	—	—
Carbonic acid	H_2CO_3	62	31	—	—
Acetic acid	$\text{HC}_2\text{H}_3\text{O}_2$	60	60	—	—
"	"	"	"	33% of $\text{HC}_2\text{H}_3\text{O}_2$	5.61 E
"	"	"	"	—	—
Tartaric acid	$\text{H}_2\text{C}_4\text{H}_4\text{O}_6$	150	75	—	—
"	"	"	"	—	—
Citric acid	$\text{H}_3\text{C}_6\text{H}_5\text{O}_7$	192	64	—	—
"	"	"	"	—	—
Oxalic acid	$\text{H}_2\text{C}_2\text{O}_4$	90	45	—	—
Hydrofluoric acid	HF	20	20	—	—
Hydrofluosilicic acid	H_2SiF_6	144	72	—	—
"	"	"	"	—	—
Hydrogen sulphide	H_2S	34	17	Saturated solution	$\frac{\text{E}}{4}$
Chlorine water	Cl_2	71	35.5	Saturated solution	$\frac{\text{E}}{5}$
Bromine	Br_2	160	80	—	—
Bromine water	"	"	"	—	—
Hydrogen peroxide	H_2O_2	34	17	—	—
"	"	"	"	—	—
Potassium hydrate	KHO	56	56	—	—
"	"	"	"	—	—
Sodium hydrate	NaHO	40	40	Sp. gr. 1.13 = 9% Na_2O	3.28 E
"	"	"	"	—	—
Ammonium hydrate	AmHO	35	35	—	—
"	"	"	"	Sp. gr. 0.96 = 10% NH_3	5.54 E

Column 6.		7.		8.
Equivalent System.		Symbol of substance taken.	Method of Preparing Reagent.	Strength of Reagent as found by trial.
Grms. per litre.	Strength.			
—	36 E	—	Sulphuric acid, sp. gr. 1.8427 at 15.5° C. Note (1).	36 E
—	5 E	—	Sulphuric acid diluted to sp. gr. 1.1527 at 15.5°. Note (2).	5 E
49	E	—	200 cc. of 5 E sulphuric acid diluted to 1 litre.	E
—	24 E —	—	Nitric acid sp. gr. 1.50. Note (3).	22.8 E
—	16 E	—	Nitric acid sp. gr. 1.4268 at 15.5° C. Note (4).	16 E
—	5 E	—	Nitric acid diluted to sp. gr. 1.1656 at 15.5° C. Note (2).	5 E
63	E	—	200 cc. of 5 E nitric acid diluted to 1 litre.	E
—	10 E	—	Hydrochloric acid sp. gr. 1.1611 at 15.5° C. Note (5).	10 E
—	5 E	—	Hydrochloric acid diluted to sp. gr. 1.0843 at 15.5° C. Note (2).	5 E
36.5	E	—	200 cc. of 5 E hydrochloric acid diluted to 1 litre.	E
—	4 E —	—	Water at 15.5° C. saturated with sulphur dioxide (sp. gr. 1.052).	3.7 E
—	$\frac{E}{10}$ —	—	Water at 15.5° C. saturated with carbon dioxide. Note (6).	—
—	17 E	—	Acetic acid solid at 10° C. Note (7).	16.9 E
—	5 E	—	294 cc. of 17 E acetic acid diluted to 1 litre. Note (2).	5 E
60	E	—	200 cc. of 5 E acetic acid diluted to 1 litre.	E
—	5 E	H ₂ C ₄ H ₄ O ₆	375 grms. dissolved and diluted to 1 litre.	5 E
75	E	"	75 " " "	E
—	5 E	H ₃ C ₆ H ₅ O ₇ , Aq	350 " " "	5 E
64	E	"	70 " " "	E
—	$\frac{3 E}{2}$	H ₂ C ₂ O ₄ , 2Aq	94.5 " " " Note (8).	$\frac{3 E}{2}$
—	12 E ?	—	Hydrofluoric acid, sp. gr. 1.15.	12.9 E
—	E	—	—	—
—	$\frac{E}{4}$ —	—	Water at 15.5° C. saturated with hydrogen sulphide.	$\frac{24 E}{100}$
—	$\frac{E}{5}$ —	—	Water at 15.5° C. saturated with chlorine.	$\frac{19 E}{100}$
—	37 E	—	Pure liquid bromine. Note (9).	—
—	$\frac{E}{2}$	—	Water at 15.5° C. saturated with bromine.	$\frac{E}{2}$
—	4 E —	—	Hydrogen peroxide, 20 volume solution.	—
—	2 E —	—	" " " " " Note (10).	—
—	5 E	KHO	280 grms. dissolved and diluted to 1 litre. Note (11).	—
56	E	"	56 " " " " " Note (12).	—
—	5 E	NaHO	200 " " " " " Note (13).	—
40	E	"	40 " " " " " Note (13).	—
—	20 E —	—	Ammonium hydrate sp. gr. 0.880. Note (14).	18.9 E
—	5 E	—	Ammonium hydrate diluted to sp. gr. 0.9643 at 15.5° C. Note (2).	5 E

Column 1.	2.	3.	4.	5. FRESSENIUS'S SYSTEM.	
Name of Reagent.	Symbol.	Molecular weight.	Equivalent weight.	Strength of Reagent. As stated.	Calculated in equivalents.
Ammonium hydrate ...	AmHo	35	35	—	—
Barium oxide	BaO	153	76.5	BaH ₂ O ₂ .8Aq. 1 to 20	$\frac{E}{3.15}$
Calcium oxide	CaO	56	28	Saturated solution	$\frac{E}{20}$
Ammonium sulphide ...	Am ₂ S	68	34	10 % NH ₃	5.54 E
"	"	"	"	—	—
Sodium sulphide	Na ₂ S	78	39	9 % Na ₂ O	3.28 E
"	"	"	"	—	—
Potassium cyanide ...	KCy	65	65	—	—
Potassium sulphate ...	K ₂ SO ₄	174	87	1 to 12	0.96 E
Potassium iodide	KI	166	166	—	—
"	"	"	"	—	—
Potassium chromate ...	K ₂ CrO ₄	194.5	97.25	—	—
Potassium metaantimoniate	KSbO ₃	209	209	Saturated solution	$\frac{E}{68}$
Potassium ferrocyanide	K ₄ FeCy ₆	368	92	1 to 12	0.79 E
Potassium ferricyanide	K ₃ Fe ₂ Cy ₁₂	658	109.7	—	—
Potassium sulphocyanate	KCyS	97	97	1 to 10	1.03 E
Sodium carbonate	Na ₂ CO ₃	106	53	2.7 to 5	3.77 E
"	"	"	"	—	—
Hydrogen disodium-phosphate	HNa ₂ PO ₄	142	47.3	1 to 10	0.84 E
Sodium acetate	NaC ₂ H ₃ O ₂	82	82	1 to 10	0.74 E
Sodium sulphite	Na ₂ SO ₃	126	63	—	—
Sodium thiosulphate ...	Na ₂ S ₂ O ₃	158	79	—	—
Sodium hypochlorite ...	NaClO	74.5	74.5	—	—
Ammonium acetate ...	AmC ₂ H ₃ O ₂	77	77	—	—
"	"	"	"	—	—
Ammonium oxalate ...	Am ₂ C ₂ O ₄	124	62	1 to 24	0.59 E
Hydrogen di-ammonium phosphate	HAm ₂ PO ₄	132	44	—	—
Ammonium carbonate ...	Am ₂ CO ₃	96	48	1 sesqui to 5 + NH ₃	5.08 E
"	"	"	"	—	—

Column 6.		7.			8.
Equivalent System.		Symbol of substance taken.	Method of Preparing Reagent.	Strength of Reagent as found by trial.	
Grms. per litre.	Strength.				
35	E	—	200 cc. of 5 E ammonium hydrate diluted to 1 litre.	E	
—	$\frac{E}{3}$	BaH ₂ O ₂ , 8Aq	52·5 grms. dissolved and diluted to 1 litre. Note (15).	—	
—	$\frac{E}{20}$	—	Water at 15·5° C. saturated with calcium hydrate.	$\frac{97 E}{2000}$	
—	5 E	—	Saturate 600 cc. of 5 E ammonium hydrate with H ₂ S in a corked flask, and then add 400 cc. of 5 E ammonium hydrate.	5 E	
34	E	—	200 cc. of 5 E ammonium sulphide diluted to 1 litre.	E	
—	5 E	—	Dissolve 200 grms. of sodium hydrate in 800 cc. water, saturate one half with H ₂ S, then add the other half and dilute to 1 litre.	5 E	
39	E	—	200 cc. of 5 E sodium sulphide diluted to 1 litre.	E	
65	E	KCy	65 grms. crystal dissolved and diluted to 1 litre.	—	
87	E	K ₂ SO ₄	87 grms. dissolved and diluted to 1 litre.	—	
166	E	KI	166 " " " Note (18).	E	
—	$\frac{E}{5}$	"	33·2 " " " Note (18).	$\frac{E}{5}$	
97·25	E	K ₂ CrO ₄	97·25 " " "	E	
3·07	$\frac{E}{08}$	K ₂ Sb ₂ O ₆ , 7Aq	Saturated solution at 15·5° C.	—	
92	E	K ₄ FeCy ₆ , 3Aq	105·5 grms. dissolved and diluted to 1 litre.	E	
109·7	E	K ₆ Fe ₂ Cy ₁₂	109·7 " " "	E	
97	E	KCyS	97 " " "	E	
—	3 E	Na ₂ CO ₃ 10Aq	429 grms. dissolved and diluted to 1 litre.	2·94 E	
53	E	"	143 " " "	0·98 E	
47·3	$\frac{2 E}{3}$ or E	HN ₃ PO ₄ , 12Aq	119·3 " " "	E	
—	4 E	NaC ₂ H ₃ O ₂ , 3Aq	544 " " "	3·96 E	
—	2 E —	Na ₂ SO ₃ , 7Aq	252 " " "	—	
79	$\frac{E}{2}$ or E	Na ₂ S ₂ O ₃ , 5Aq	124 " " "	—	
—	—	—	—	—	
—	5 E	—	294 cc. of 17 E acetic acid, neutralised with strong ammonium hydrate and diluted to 1 litre.	5 E	
77	E	—	200 cc. of 5 E ammonium acetate diluted to 1 litre.	E	
37·2	$\frac{3 E}{5}$	Am ₂ C ₂ O ₄ , Aq	42·6 grms. dissolved and diluted to 1 litre. Note (16).	$\frac{3 E}{5}$	
44	$\frac{2 E}{3}$ or E	HAm ₂ PO ₄	44 grms. dissolved and diluted to 1 litre.	E	
—	5 E	—	196·7 grms. of ammonium sesquicarbonate dissolved in 333·3 cc. of 5 E ammonium hydrate and diluted to 1 litre.	—	
48	E	—	200 cc. of 5 E ammonium carbonate solution diluted to 1 litre.	—	

Column 1. Name of Reagent.	2. Symbol.	3. Mole- cular weight.	4. EQUIVA- lent weight.	5. FRESSENIUS'S SYSTEM.	
				Strength of Reagent.	
				As stated.	Calculated in equiva- lents.
Hydrogen ammonium carbonate	HAmCO ₃	79		—	—
Ammonium chloride ...	AmCl	53'5	53'5	1 to 8	2'34 E
" "	" "	"	"	—	—
Ammonium sulphate ...	Am ₂ SO ₄	132	66	—	—
Barium chloride	BaCl ₂	208	104	1 to 10	0'82 E
Barium nitrate	BaN ₂ O ₆	261	130'5	1 to 15	0'51 E
Barium carbonate	BaCO ₃	197	98'5	—	—
Strontium sulphate ...	SrSO ₄	183'5	91'75	—	—
Calcium chloride	CaCl ₂	111	55'5	1 cryst. to 5	1'83 E
Calcium sulphate	CaSO ₄	136	68	Saturated solution	$\frac{E}{30}$
Magnesium chloride ...	MgCl ₂	95	47'5	—	—
Magnesium sulphate ...	MgSO ₄	120	60	1 to 10	0'81 E
Ferrous sulphate	FeSO ₄	152	76	—	—
Ferric chloride	Fe ₂ Cl ₆	325	54'17	—	—
Plumbic acetate	PhC ₄ H ₆ O ₄	325	162'5	1 to 10	0'53 E
Plumbic nitrate	PhN ₂ O ₆	331	165'5	—	—
Argentio nitrate	AgNO ₃	170	170	1 to 20	0'30 E
" " " "	" " " "	" " " "	" " " "	—	—
Argentio sulphate	Ag ₂ SO ₄	312	156	—	—
Mercurous nitrate	Hg ₂ N ₂ O ₆	524	262	—	—
Mercuric chloride	HgCl ₂	271	135'5	1 to 16	0'46 E
Cupric sulphate	CuSO ₄	159'5	79'75	1 to 10	0'80 E
Cupric chloride	CuCl ₂	134'5	67'25	—	—
Stannous chloride	SnCl ₂	189	94'5	—	—
Auric chloride	AuCl ₃	303'1	101	1 to 30	0'33 E
Platinic chloride	PtCl ₄	339'1	84'8	—	0'77 E
Magnesia mixture (for phosphoric acid)	—	—	—	—	—

Column 6.		7.		8.
Equivalent System.		Symbol of substance taken.	Method of Preparing Reagent.	Strength of Reagent as found by trial.
Grms. per litre.	Strength.			
—	$\frac{3 \text{ E}}{2}$ or 3 E	—	A saturated solution made by passing excess of carbon dioxide into 3 E ammonium hydrate.	—
—	5 E	AmCl	267.5 grms. dissolved and diluted to 1 litre.	5 E
53.5	E	"	53.5 " " "	E
66	E	Am ₂ SO ₄	66 " " "	E
164	E	BaCl ₂ , 2Aq	122 " " "	E
65.25	$\frac{\text{E}}{2}$	BaN ₂ O ₆	65.25 " " " Note (17).	$\frac{\text{E}}{2}$
—	2 E	BaCO ₃	197 grms. freshly precipitated, suspended in water and diluted to 1 litre.	—
0.153	$\frac{\text{E}}{600}$ —	SrSO ₄	Water at 15.5° C. saturated with precipitated strontium sulphate.	—
55.5	E	CaCl ₂ , 6Aq	109.5 grms. dissolved and diluted to 1 litre.	0.97 E
2.27	$\frac{\text{E}}{30}$	CaSO ₄ , 2Aq	Water at 15.5° C. saturated with precipitated calcium sulphate.	—
47.5	E	MgCl ₂ , 6Aq	101.5 grms. dissolved and diluted to 1 litre.	0.98 E
60	E	MgSO ₄ , 7Aq	123 grms. dissolved and diluted to 1 litre.	E
76	E	FeSO ₄ , 7Aq	139 " " "	0.97 E
54.17	E	—	18.67 grms. of iron, as Fe ₂ H ₆ O ₆ , dissolved in 200 cc. of 5 E hydrochloric acid and diluted to 1 litre.	—
162.5	E	PbC ₄ H ₆ O ₄ , 3Aq	189.5 grms. dissolved and diluted to 1 litre.	E
165.5	E	PbN ₂ O ₆	165.5 " " "	0.98 E
170	E	AgNO ₃	170 " " " Note (18).	E
—	$\frac{\text{E}}{5}$	"	34 " " " Note (18).	$\frac{\text{E}}{5}$
7.8	$\frac{\text{E}}{20}$	Ag ₂ SO ₄	Water at 15.5° C. saturated with freshly precipitated argentic sulphate.	$\frac{\text{E}}{21}$
52.4 +	$\frac{\text{E}}{5}$ +	Hg ₂ N ₂ O ₆ , 2Aq	56 grms. dissolved in 40 cc. of 5 E nitric acid and diluted to 1 litre, a little mercury being placed in a bottle.	$\frac{\text{E}}{5}$
54.2	$\frac{2 \text{ E}}{5}$	HgCl ₂	54.2 grms. dissolved and diluted to 1 litre.	$\frac{2 \text{ E}}{5}$
79.75	E	CuSO ₄ , 5Aq	124.75 " " "	E
67.25	E	CuCl ₂ , 2Aq	85.25 " " "	0.95 E
94.5 +	E +	SnCl ₂ , 2Aq	112.5 grms. dissolved in 200 cc. of 5 E hydrochloric acid and diluted to 1 litre, a little tin being placed in bottle.	0.96 E
20.2	$\frac{\text{E}}{5}$	—	13.1 grms. of metallic gold, converted into auric chloride, dissolved and diluted to 1 litre. Note (18).	—
84.8	E	—	49.3 grms. of metallic platinum, converted into platmic chloride, dissolved and diluted to 1 litre. Note (18).	—
—	E	—	Dissolve 68 grms. MgCl ₂ , 6 Aq in about 500 cc. of water, add 165 grms. AmCl, then 300 cc. of 5 E ammonium hydrate, and dilute to 1 litre.	—

(7) Acetic acid, anhydrous, sp. gr. 1.0635, containing 100 per cent., by calculation gives 17.7 E.

Acetic acid, sp. gr. 1.0598, solid at 50° F., as supplied, titrated, gave 16.9 E.

(8) Oxalic acid. A $\frac{3}{2}$ E solution does not crystallise out at 15° C., but does so at 10° C.

(9) Bromine, sp. gr. 2.966, by calculation gives 37.08 E.

(10) Hydrogen peroxide, 20 volumes, by calculation gives 3.6 E.

(11) Hydrogen peroxide, 10 volumes, by calculation gives 1.8 E.

(12) Potassium hydrate, about one-tenth more than the quantity given in the table is required, owing to most samples containing water.

(13) Sodium hydrate prepared from sodium is employed.

(14) Ammonium hydrate, sp. gr. 0.88, containing 38 per cent., by calculation gives 19.7 E.

Ammonium hydrate, sp. gr. 0.88, as supplied, three samples titrated gave 18.0 E, 16.2 E, and 18.9 E.

(15) Barium hydrate. The strength of a *saturated* solution varies considerably with the temperature.

(16) Ammonium oxalate. A stronger solution than $\frac{3}{5}$ E crystallises out at the ordinary temperature.

(17) Barium nitrate is not sufficiently soluble to make an E solution.

(18) Where reagents are expensive an $\frac{E}{5}$ or $\frac{E}{10}$ solution may be made for qualitative tests.

It will be seen from the Table that a large number of the reagents are made of E strength. Experience, however, has shown that there are cases in which it is necessary to employ certain reagents, and especially the acids, stronger than this; consequently a number of 5 E reagents have been introduced, 5 being a convenient figure for calculations on the decimal system. These are more accurately made, and keep more

constant in strength than the concentrated reagents, which are liable to a variation of about one equivalent.

About three-fourths of the reagents given in the Table may be kept in well-stoppered bottles for a considerable time without appreciable change. In the few remaining cases where the variation in strength is considerable, the unwelcome fact is expressed in column 6 of the preceding Table and on the reagent bottles in the following manner:—

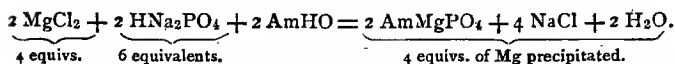
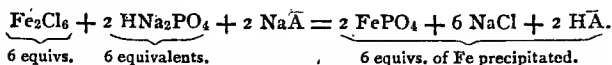
Where it is known that the reagent has been made the standard strength, but becomes weaker on keeping, it may be expressed by the sign — placed after the symbol E, thus:—4 E — Sulphurous Acid. If, on the other hand, there is an increase of strength, as in the case of the acid solution of stannous chloride kept in contact with metallic tin, it may be expressed thus:—E + Stannous Chloride. The analyst is thus forewarned as to any irregularity which may occur in the strength of such reagents.

The practical application of these multiple and fractional equivalents is well illustrated by the chlorine and bromine reagents given in the table. Pure liquid bromine is shown as 37 E; a saturated solution of bromine in water as $\frac{E}{2}$; and a saturated solution of chlorine in water $\frac{E}{5}$. Now if we know that 1 cc. of liquid bromine (37 E) is required to effect a definite amount of oxidation in a solution, we see at a glance by means of these equivalents that it would require 74 cc. of $\frac{E}{2}$ bromine water or 185 cc. of $\frac{E}{5}$ chlorine water to effect the same *quantitative* result.

It may here be pointed out that a few reagents require a double index of their strength, e.g., E or $\frac{2E}{3}$ hydrogen disodium phosphate.* This reagent behaves as an E solution if employed to precipitate iron as phosphate, but only as $\frac{2E}{3}$ when

* On this account the second or third label previously given is preferred.

employed to precipitate magnesium as ammonium magnesium phosphate owing to ammonium taking part in the reaction. A reference to the chemical equations representing the reactions will illustrate this:—



Having thus briefly described the proposed system of reagents, we will proceed to notice some of the advantages attending its employment in practical and analytical chemistry:—

1. It affords the most convenient method of expressing the *proportionate* strength or precipitating power of each reagent, and one which may be plainly indicated on each bottle.

2. It indicates as near as can be practically attained the *actual* strength of each reagent.

Thus:—36 E sulphuric acid contains 36 m.grm. equivalents in 1 cc., and knowing this we can readily calculate the quantity of any constituent, *e.g.*, the equivalent weight of sulphuric acid being 49, we have $49 \times 36 = 1,764$ m.grms. = 1.764 grms. of sulphuric acid present in 1 cc. Similarly, each cc. contains $1 \times 36 = 36$ m.grms. = 0.036 gm. of hydrogen, $16 \times 36 = 576$ m.grms. = 0.576 gm. of sulphur, and $32 \times 36 = 1,152$ m.grms. = 1.152 grms. of oxygen.

3. By observing the quantity of a reagent employed in an analysis, we can calculate the quantity of by-product therefrom, a point of considerable importance in deciding upon the dilution of our solutions.

For example:—If, during a quantitative analysis, 10 cc. of 36 E sulphuric acid is added and afterwards neutralised by sodium carbonate, we observe that 10×36 equivalents of sodium sulphate will be produced. Now, since the equivalent of sodium sulphate is 71, we have $71 \times 36 \times$

10 = 25,560 m.grms. = 25.56 grms. as the quantity of anhydrous sodium sulphate formed by the reaction. Knowing this, we are forewarned to dilute sufficiently before proceeding with the analysis.

4. In qualitative analysis we are able to form a better judgment of the amount of substance present than when using the reagents of indefinite strength hitherto employed.

5. The strength of reagents employed in chemical investigations may be most conveniently expressed on this system.

For example :—

Nitric acid sp. gr. 1.4268 at 15.5° C. is *exactly* 16 E in strength.

"	"	1.1656	"	"	5 E	"
"	"	1.0345	"	"	E	"

Now it is evident that the latter mode of expression is far simpler, and gives a much more accurate idea of the strength of the acid than does its specific gravity. These equivalent numbers would be a valuable addition to sp. gr. tables.

6. Reagents may be most readily diluted to the various strengths required in chemical analysis.

For example :—Nitric acid sp. gr. 1.42 = 16 E approximately may be readily obtained by distillation. To make an acid of E strength, it is simply necessary to dilute 1 cc. of 16 E acid with water to the volume of 16 cc. To make 2 E acid, dilute 2 cc. of 16 E acid to 16 cc. ; or to make 15 E acid, take 15 cc. of 16 E acid and dilute to 16 cc., and so on. It is not usually necessary to take changes of temperature into consideration, except in the case of strong sulphuric acid.

7. This system affords the simplest method of calculating the theoretical quantity of reagent required to effect any chemical change.

For example :—If we wish to find the quantity of reagent necessary to precipitate 1 grm. of calcium from its solution ; we already know that 1 cc. of E reagent, say E ammonium carbonate, will precipitate 20 m.grms. (1 m.grm.

equivalent); consequently, 50 cc. of E reagent will be required to precipitate 1 gm. If preferred, 10 cc. of 5 E ammonium carbonate may be used instead. In like manner we can calculate the quantity of acid theoretically required to *dissolve* a given quantity of metal or other substance. Thus, 1 gm. of zinc, containing 30.8 m.grms. equivalents, will theoretically require 30.8 cc. of E acid or 6.16 cc. of 5 E acid to effect its solution. In the same way it may be shown that while to dissolve or precipitate 1 gm. of thallium theoretically requires only 4.9 cc. of *E reagent*, to dissolve or precipitate 1 gm. of beryllium would require 217.4 cc. of *E reagent*.

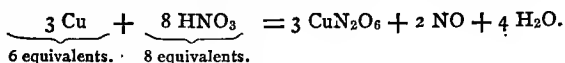
The following table gives the number* of m.grm. equivalents contained in 1 gm. of each of the more common elementary substances. This enables us to find at a glance the number of cc. of E. reagent theoretically required to effect the solution or precipitation of 1 gm. of each substance.

Symbol of Element.	Atomic Weight.	Equivalent Weight.	M.grm. equivs. per gm., or cc. of E.
Al'''	27	9	111.1
Sb'''	120	40	25
Sb ^v	"	24	41.7
As'''	75	25	40
As ^v	"	15	66.7
Ba''	137	68.5	14.6
Be''	9.2	4.6	217.4
Bi'''	210	70	14.3
Bi ^v	"	42	23.8
B'''	11	3.67	272.5
Br'	80	80	12.5
Cd''	112	56	17.9
Ca''	40	20	50
C''	12	6	166.7
Ci ^v	"	3	333.3
Cl'	35.5	35.5	28.2

* This number is the reciprocal of the equivalent weight $\times 1,000$; consequently a table of reciprocals would be applicable for all substances, and might be used for this purpose.

Symbol of Element.	Atomic weight.	Equivalent weight.	M.grm. equivs. per grm., or cc. of E.
Cr ⁱⁱⁱ	52.5	17.5	57.1
Cr ^{vi}	"	8.75	114.3
Co ⁱⁱ	59	29.5	33.9
Co ⁱⁱⁱ	"	19.67	50.8
Cu ⁱ	63.5	63.5	15.7
Cu ⁱⁱ	"	31.75	31.5
F ⁱ	19	19	52.6
Au ⁱ	196.6	196.6	5.1
Au ⁱⁱⁱ	"	65.53	15.3
H ⁱ	1	1	1000.0
I ⁱ	127	127	7.9
Fe ⁱⁱ	56	28	35.7
Fe ⁱⁱⁱ	"	18.67	53.6
Pb ⁱⁱ	207	103.5	9.7
Li ⁱ	7	7	142.9
Mg ⁱⁱ	24	12	83.3
Mn ⁱⁱ	55	27.5	36.4
Mn ^{iv}	"	13.75	72.7
Mn ^{vi}	"	9.17	109.1
Hg ⁱ	200	200	5
Hg ⁱⁱ	"	100	10
Ni ⁱⁱ	59	29.5	33.9
Ni ⁱⁱⁱ	"	19.67	50.8
P ⁱⁱⁱ	31	10.33	96.8
P ^v	"	6.2	161.3
Pt ⁱⁱ	194.381	97.19	10.3
Pt ^{iv}	"	48.59	20.6
K ⁱ	39.1	39.1	25.6
Si ^{iv}	28	7	142.9
Ag ⁱ	108	108	9.3
Na ⁱ	23	23	43.5
Sr ⁱⁱ	87.5	43.75	22.9
S ⁱⁱ	32	16	62.5
Si ^v	"	8	125
S ^{vi}	"	5.33	187.6
Sn ⁱⁱ	118	59	16.9
Sn ^{iv}	"	29.5	33.9
Tl ⁱ	204	204	4.9
Tl ⁱⁱ	"	68	14.7
Ti ⁱ	50	25	40
Ti ^{iv}	"	12.5	80
Zn ⁱⁱ	65	32.5	30.8

Should a secondary reaction occur, as for instance in the action of copper upon nitric acid, the quantity of reagent required may be deduced from the equation representing the chemical change, as follows:—



Here we see that 6 equivalents of copper require 8 equivalents of nitric acid, or one-third more than the normal quantity; therefore $1\frac{1}{3}$ times the quantity of acid, as obtained from the table, will be required.

8. The system further affords a simple method of calculating the quantity of gas given off in chemical reactions.

Since 1 cc. of an E reagent, in acting upon excess of metal, will liberate a m.grm. equivalent of hydrogen = 11.16 cc., the volume given off by x cc. of any E reagent will be $x \cdot 11.16$ cc. Conversely, we may calculate the quantity of acid required to produce a given volume of hydrogen.

9. In working with this system we have the most convenient method of expressing the precise acidity, &c., of chemical solutions intended for accurate titration, precipitation, &c.

For example:—In Margueritte's method of estimating iron volumetrically by means of *standard* $\frac{\text{E}}{10}$ potassium permanganate, I recommend that the solution for titration be acidified to E or $\frac{\text{E}}{2}$ sulphuric acid, thus expressing the acidity at which it is preferred the reaction should take place.

An example of the application of these reagents to the *separation of metals* may be given. It is known that a solution of zinc chloride containing 1 gram. of zinc. in 100 cc. requires the presence of an appreciable quantity of free hydrochloric acid to prevent the precipitation of the *zinc* by hydrogen sulphide. It is also known that *lead* is not completely precipitated from its solutions by hydrogen sulphide if more than 2.5 per cent. of free hydrochloric acid be present. The question arises how to

acidify the solution so as to allow of the complete precipitation of the *lead* without the co-precipitation of the *zinc*. I have found that *lead* can be completely precipitated by hydrogen sulphide from an $\frac{E}{2}$ hydrochloric acid solution without perceptible co-precipitation of *zinc*, and now by acidifying the solution to $\frac{E}{2}$ hydrochloric acid, I always secure the most favourable conditions for the separation of these metals.

As illustrating the application of this system to *precipitation*, I may mention the determination of *lead as sulphate*. It is well known that lead sulphate is soluble in strong sulphuric acid, and also in pure water, but that in dilute sulphuric acid it is practically insoluble. The strength of sulphuric acid corresponding to the least solubility does not appear to have been recorded. I have, however, found that lead sulphate is least soluble in *E sulphuric acid*, and now always adopt this strength in my analytical work.

As a final illustration we will take the precipitation of phosphoric acid by magnesia mixture.

David Lindo, in *Chemical News*, vol. xlviii., p. 217, has given the conditions under which he considers this precipitation can be best effected. I have calculated the quantities of reagent used in terms of *E*, and the conditions of precipitation may be stated as follows :—

1st. Solution to be neutral or slightly ammoniacal.

2nd. Dilution of P_2O_5 to be from $\frac{E}{5}$ to $\frac{E}{20}$, or about 1 grm. in from 200 to 800 cc.

3rd. Dilution of $AmCl$ to be from 0 to $\frac{E}{2}$.

4th. After precipitate has settled, add $\frac{1}{3}$ bulk of 5 *E* ammonium hydrate.

5th. Wash with $\frac{3}{2} E$ ammonium hydrate.

6th. Magnesia mixture up to $\frac{E}{3}$ in excess does not vitiate result.

7th. Magnesia mixture counteracts solvent action of ammonium chloride.

Preparation of Standard Equivalent Solutions.—In volumetric analysis, where the measure of a certain substance is to be obtained from the volume of a certain reagent required to neutralize or precipitate it, it is essential that its value should be *accurately* known, so that a definite volume of it will be equivalent to, or a measure of, any substance which it is desired to determine. The following method used for preparation of standard solutions in alkalimetry, acidimetry, &c., will suffice to illustrate how *Standard* equivalent solutions are prepared.

Preparation of Standard E Sodium Carbonate.—Since sodium carbonate can be obtained in a pure state, and any moisture it may contain be driven off by gentle ignition, it can be used as a basis for the standardization of all acid and alkaline solutions. It is prepared by dissolving 53 grms. of pure anhydrous sodium carbonate in water and diluting to 1,000 cc. at 15° C. About 56 grms. of the carbonate are weighed out into a platinum dish, and gently ignited to a dull red heat until all moisture has been eliminated; it is cooled in a desiccator, and the 53 grms. quickly weighed from it.

Preparation of Standard E Sulphuric Acid.—Standard E sulphuric acid should contain 49 grms. real H_2SO_4 in a litre of water.

The following will illustrate how this is obtained:—29 cc. of strong sulphuric acid of sp. gr. 1.84 was dissolved in water, and diluted to 1,000 cc. at 15° C. 30 cc. of standard E Na_2CO_3 was run from a burette into a 100 cc. beaker, and two drops of methyl orange (1 in 1,000) added, and diluted to 50 cc. A burette was filled with the H_2SO_4 prepared as above, and allowed to gradually run into the yellow-tinted alkaline solution, stirring, until the last drop added produced a pink coloration, when 28 cc. were required instead of 30 cc. Now it is necessary to add water to the acid, since it is too strong, to an extent that will make it exactly E, and every 28 cc. of the acid will

want diluting to 30 cc. There was 910 cc. of acid left; therefore $\frac{910}{28} = 32.5$ times; and $32.5 \times 2 = 65$ cc. more water to be added; on adding this quantity, and again titrating as above, 30 cc. of alkali required exactly 30 cc. of acid, and therefore, the solutions were chemically equivalent to each other.

Preparation of Standard E Sodic Hydrate.—A Standard E solution of sodic hydrate should contain 40 grms. of NaHO in a litre, but since pure sodic hydrate always contains some water, a quantity more than this must be taken. 42 grms. of pure sodic hydrate prepared from sodium was dissolved in water and diluted to 1,000 cc. at 15° C., and on titrating 30 cc. with the above standard E acid, exactly 30 cc. were required for neutralization; but often it requires more acid than this, then it must be diluted until it is of the right strength, in a similar manner to that adopted for the dilution of the sulphuric acid.

PART I.

METALS, ALLOYS, ETC.

METHOD FOR THE COMPLETE ANALYSIS OF COPPER.

Insoluble Residue and Stock Solution.—Weigh out 50 grms. of the sample in turnings, and transfer to a $1\frac{1}{2}$ litre beaker, pour on 600 cc. of nitric acid, sp. gr. $1.2 = 6.15$ E, and allow to digest on a warm plate until all that is soluble is dissolved; evaporate now over water bath until crystals of copper nitrate come out. Dissolve in 800 cc. of distilled water and filter off any residue through a small tared filter paper; detach any portion adhering to the sides of the beaker with the aid of a feather and wash till free from the copper salt with E HNO_3 , and finally with water; dry the filter at 100°C ., and weigh between watch glasses. If the residue is at all considerable it is to be kept and fused with sodic sulphide, and treated by processes herein described. There will be a separate method adopted for the estimation of tin. The filtrate is now diluted to 1,000 cc. at 15.5°C ., and poured into a dry stoppered bottle, from which known volumes are to be taken for the estimation of most of the impurities.

Estimation of Fe, Ni, Co, Zn.—200 cc. (equivalent to 10 grms. of sample) of the stock solution, are measured out accurately, and poured into a litre beaker; add now 20 cc. of sulphuric acid, sp. gr. $1.84 = 36$ E, and evaporate as far as possible on the water bath and finally on the sand bath until all fumes of H_2SO_4 are eliminated. Allow to cool and

digest with 300 cc. of $\frac{E}{2}$ H_2SO_4 , until all that is soluble is dissolved, decant as much as possible of the clear blue solution into a tared $1\frac{1}{2}$ litre flask, provided with a cork, and pass the residual solution containing any $PbSO_4$, &c., through a filter paper and finally wash with $\frac{E}{2}$ H_2SO_4 until free from copper salt—reject residue. To the solution add 50 cc. of strong $HCl = 10 E$, and dilute to 1,000 cc., the solution being now .65 E total free acid. Heat the solution to $70^\circ C$. and saturate with sulphuretted hydrogen, after which the cork is introduced, allowed to cool to ordinary temperature, and the whole weighed. Deduct from this the weight of the dried flask, + 15.07 grms. CuS (the amount of CuS formed by the 10 grms. of sample taken; this is not strictly speaking correct, but there is no practical error introduced for ordinary coppers) and the amount of liquid present will be obtained. Decant through a filter carefully and as quickly as possible (to prevent oxidation) into another tared $1\frac{1}{2}$ litre flask and weigh; and calculate from this the amount of sample equivalent to the volume decanted (*vide* example, page 34). The liquid is now poured out of the flask into a beaker and evaporated to complete dryness over the sand bath. Dissolve the residue in 20 cc. of $\frac{1}{2} E$ HCl , pour into a small, tall beaker and saturate with SH_2 , and filter off any little CuS , &c., that was not completely separated in the first instance. Neutralize the solution with 20 E $AmHO$, add $\frac{1}{2}$ cc. in excess, and saturate with SH_2 . Allow to settle in a warm place for about a quarter of an hour and filter off the precipitated sulphides of Fe, Ni, &c.; wash quickly with $\frac{1}{2} E$ Am_2S solution and dissolve residue in 3 cc. warm 5 E HCl , with addition of a crystal of chlorate of potash: boil the solution carefully until all free chlorine has escaped, add 4 cc. of 5 E $AmCl$, dilute to about 20 cc., and add, very cautiously, 5 E solution of ammonic carbonate, until nearly all the free acid is neutralized; then add, drop by drop, a solution of E ammonic carbonate, until a slight turbidity is produced. Heat slowly to boiling, cool, and add 3 cc.

of 5 E AmHO ; allow to stand a short time, filter through a small filter paper, wash with $\frac{1}{2}$ E AmCl, reserve filtrate, re-dissolve in 3 cc. of 5 E HCl, and re-precipitate the iron now as ferric hydrate $[\text{Fe}_2(\text{HO})_6]$ with 1 cc. of ammoniac hydrate '880 sp. gr. = 20 E ; heat to boiling, and filter off through one of Schleicher & Schüll's smallest size chemically pure filter papers, leaving less than '0001 grm. of ash on incineration, a quantity which may be disregarded in the determination. (It may be here remarked that throughout the copper analysis, where such small quantities are to be weighed, it will be advisable to use these papers. They can be obtained from Messrs. Townson & Mercer, London, who are the sole agents.) Wash the precipitate with water, until the washings no longer give a turbidity with nitrate of silver, mix filtrate and reserve, spread the filter on to a watch glass and dry in the water oven. Scrape as much as possible of the oxide into a small tared porcelain crucible and ignite the filter paper separately, adding the residue to the main portion. The crucible and its contents are now gradually ignited to a full red heat in the oxidising flame of a Bunsen or in a good muffle, after which it is cooled in a good desiccator and weighed. The increase in weight of the crucible is the amount of iron originally present in 10 grms. of the sample now in the state of peroxide (Fe_2O_3). This weight, multiplied by '700, and divided by amount of sample taken, $\times 100$, will give the percentage of iron in the sample.

The mixed filtrates, containing the zinc, &c., are now evaporated to 30 cc. and made just acid with HCl, and then *just* alkaline with E Na_2CO_3 ; add 4 drops of 5 E HCl, saturate with SH_2 , filter off any zinc sulphide if present, and wash. Boil the solution until SH_2 has gone off, neutralize with E Na_2CO_3 , make $\frac{\text{E}}{5}$ with acetic acid, add 2 cc. E acetate of soda, heat to 70°C ., saturate with SH_2 , allow precipitate to subside, filter off the nickel sulphide quickly, and wash with SH_2 water (test the solution for manganese by neutralizing with 20 E AmHO, and adding 5 cc. 5 E Am_2S). Dissolve off the filter with 5 cc. of 10 E HCl, with addition of a crystal of potassic

chlorate, boil till chlorous odour has gone, and wash out into a porcelain dish; volume being here now 15 cc., neutralize with 2 E Na_2CO_3 , boil, and add 2 cc. of 2 E NaHO solution (pure) to the hot solution, and boil for a few minutes: filter off hydrate of nickel, wash with boiling water till free from alkali, dry in water oven, scrape precipitate into a tared crucible, ignite the filter separately, and add residue to main portion: ignite the crucible at a red heat for ten minutes, cool and weigh the NiO , which $\times .7867$ and \div the amount of sample taken, $\times 100 =$ percentage of nickel in the sample.

Test the nickel oxide for cobalt by treating the residue in the crucible with $\frac{1}{2}$ cc. of aqua regia, evaporating to dryness, dissolving in 5 cc. of water, adding a drop of 5 E acetic acid and 2 cc. of 4 E potassic nitrite solution, and allowing to stand at 50°C . for some hours, when, if cobalt is present, a yellow precipitate of double nitrite of cobalt and potassium will be produced.

Estimation of Antimony and Arsenic (*Abel's process*).—Take 200 cc. of stock solution, equivalent to 10 grms. of original sample, and pour into a litre beaker. Add 5 cc. of an E solution of nitrate of lead $[\text{Pb}(\text{NO}_3)_2]$, 70 cc. of 5 E AmHO , and 10 cc. 5 E Am_2CO_3 . Dilute to 800 cc., and allow to stand twenty-four hours with frequent stirring, after which it is allowed to stand without stirring, until the precipitate of arseniate, antimoniate, carbonate, &c., of lead has settled to the bottom. Decant as much as possible of the clear solution, filter off the remaining solution, wash the precipitate with 2 E AmHO till free from copper salt, &c., spread out the filter on a glass plate, carefully remove as much as possible of the precipitate into an 80 cc. beaker with a platinum spatula, and remove any precipitate adhering to the paper with 30 cc. of $1\frac{1}{2}$ E oxalic acid, with the aid of a wash bottle. Boil the solution for half an hour, filter off, and wash with water. The antimony and arsenic is now in solution. Dilute filtrate to 150 cc., neutralize with 2 E pure solution of sodic hydrate, add 7 grms. by weight in excess, saturate with sulphuretted hydrogen (SH_2), allow to stand in a warm place for some time, filter off sulphide of

lead, &c., and wash with $\frac{1}{2}$ E Na_2S solution. Neglect residue, dilute filtrate to 225 cc., carefully add 20 cc. of 10 E HCl , allow the precipitate of antimony and arsenic sulphides, and free sulphur, to settle in a warm place for a couple of hours, filter off, wash with SH_2 water, dry in water oven, half fill a small tall beaker with bisulphide of carbon, fold the paper up with its contents, place it cone downwards into the CS_2 , and cover with a watch glass; by this means the great bulk of the free sulphur is dissolved out.

After digesting for a couple of hours the filter is spread out on a watch glass and dried, the precipitate brushed into a small beaker and dissolved in a mixture of 2 cc. 22 E HNO_3 (fuming) and 6 cc. 10 E HCl at a gentle heat. Dilute with water to 50 cc., add 4 grms. of tartaric acid previously dissolved in 5 cc. of water, neutralize with 5 E AmHO and add 4 cc. in excess. Add 10 cc. of E magnesia mixture (prepared by dissolving 68 grms. magnesian chloride and 165 grms. AmCl in 290 cc. of 5 E AmHO and diluting to 1 litre) and dilute to 100 cc., stir well and allow to stand twenty-four hours, filter off the arseniate of magnesia and ammonia, detaching the last portions of the precipitate from the beaker with the aid of some of the filtrate and a feather. This is adopted in order to minimize the number of washings with the 2 E AmHO in which the precipitate is somewhat soluble. Wash with 2 E AmHO , and note the total volume of the solution, which should be about 120 cc., and reserve. Dry the precipitate in the water oven, brush into a tared porcelain or platinum crucible, saturate the paper with E ammonium nitrate solution, dry, ignite by means of platinum wire and Bunsen, and add the residue to main portion. Ignite *gradually*, until the crucible is at a bright red heat, cool, and weigh. The increase of weight of the crucible is due to pyroarsenate of magnesia ($\text{Mg}_2\text{As}_2\text{O}_7$). Add to this weight .0032 gram. due to the solubility of the arseniate of magnesia and ammonia in the 120 cc. of the ammoniacal solution, multiply this weight by $.4839 \times 10$, and this will give the percentage of arsenic in the sample.

The filtrate containing the antimony is now neutralized with

10 E HCl and 5 cc. added in excess. Saturate with SH_2 , allow the sulphide of antimony to settle in a warm place, filter off, wash with SH_2 water, dry in water oven, scrape off paper into a small tared crucible, moisten the paper with E AmNO_3 solution, dry and ignite, and add residue to crucible; add 8 to 10 times its bulk of 22 E HNO_3 , cover with a watch glass and digest for some time at a gentle heat; evaporate off excess of acid and carefully ignite at a red heat for ten minutes, cool in desiccator and weigh the Sb_2O_4 . Multiply this weight by $\cdot 7922 \times 10$, and this will give the percentage of Sb present in sample.

Estimation of Lead and Bismuth (*Abel's process*).—Take 200 cc. of the stock solution (= 10 grms. of sample) and pour into a litre beaker. Now add 20 cc. of E sodic phosphate solution, dilute to 650 cc., add 60 cc., 20 E AmHO , stir well, and allow to stand for forty-eight hours. Decant as much as possible of the clear liquor, filter the residue on a small filter paper, wash till free from copper salts with 2 E AmHO , and dissolve the phosphates of lead, bismuth, &c., on the filter into a small beaker with 5 cc. of warm 10 E HCl, washing out with hot water. Let volume here be now 20 cc., neutralize with 20 E AmHO , add 1 cc. in excess, saturate with SH_2 gas, allow the precipitated sulphides of lead, bismuth, &c., to subside in a warm place, filter and wash with SH_2 water, place a test tube under the funnel and pour on to the precipitate 5 cc. of 8 E HNO_3 (warm), passing it through several times.

There are invariably lumps of dark sulphur containing traces of lead and bismuth left on the paper. To separate the sulphur, push the sulphur particles as far as possible down to the bottom of the filter, dry in water oven, and digest in some carbon bisulphide contained in a small beaker until the sulphur is dissolved out; now dry, treat with 8 E HNO_3 , and add this to the main portion. The filter is dried, moistened with ammonium nitrate, carefully incinerated, and the ash likewise added to main solution. The whole is boiled until all is dissolved. Dilute to 15 cc., nearly neutralize solution with

AmHO, using 20 E at first, and towards the end a 5 E solution, drop by drop, until the precipitate which is formed goes into solution with difficulty, and, the solution being perfectly clear, add now some recently precipitated copper hydrate, $\text{Cu}(\text{HO})_2$, in slight excess with the aid of a glass spatula, stir well, and allow to stand a couple of hours with frequent stirring. (The copper hydrate is prepared by diluting 5 cc. of E CuCl_2 to 80 cc., and adding, gradually stirring, 15 cc. of $\frac{\text{E}}{2}$ NaHO solution, in the cold, filtering, and washing with water till free from NaHO.) This quantity of $\text{Cu}(\text{HO})_2$ will generally be found sufficient for ordinary coppers. The bismuth is thus precipitated as $\text{Bi}(\text{HO})_3$, and the lead left in solution as nitrate. Filter off and wash with hot water, and reserve filtrate for the estimation of lead. Dissolve off the $\text{Bi}(\text{HO})_3$ and excess of $\text{Cu}(\text{HO})_2$ in 3 cc. of 5 E HNO_3 , wash the paper with $\frac{\text{E}}{5}$ HNO_3 , dilute to 20 cc, saturate with SH_2 , allow to settle, filter off sulphides, dissolve in 8 E HNO_3 as before, dilute to 15 cc., neutralize with 5 E Am_2CO_3 , add 5 cc. in excess, and allow to digest on a hot plate for some time; filter off the bismuth carbonate, wash, dry in water oven, brush off to a small watch glass, ignite the filter carefully, and add the ash to a tared crucible. Digest this in a few drops of 16 E HNO_3 , evaporate, add main portion of precipitate from watch glass, and ignite the whole cautiously in an oxidizing flame of a Bunsen burner for ten minutes, cool and weigh. Increase in weight = $\text{Bi}_2\text{O}_3 \times .8965 \times 10 =$ percentage of bismuth present in sample.

[This process is liable to give low results for lead. The lead could be estimated in the portion taken for the iron, &c., estimation, but the residue should be taken up in 5 E sulphuric acid, to take bismuth sulphate in solution, and then diluted five times with water before filtering off the lead sulphate.]

Estimation of Combined Oxygen.—Clean a piece of the sample 2 in. \times 1½ in. \times ½ in. by immersing for a few seconds

in 16 E HNO_3 ; wash with distilled water, then with alcohol, dry quickly in water oven and weigh. Immerse in 150 cc. of E AgNO_3 solution for three hours, occasionally scraping off the metallic silver, &c., from the surface of the copper with a stir rod. (Reaction with regard to the combined oxygen = $\text{Cu}_2\text{O} + 2 \text{AgNO}_3 = \text{Ag}_2 + \text{Cu}(\text{NO}_3)_2\text{CuO}$.) The portion of the sample unacted upon is taken out and washed with the aid of a jet of water from a wash bottle, and a feather; the water is taken off with alcohol, and the sample dried in water oven, cooled, and weighed. The difference between this and the original weight is the amount of sample taken for the estimation.

The liquid and washings are decanted through a filter paper, and the precipitated silver and basic nitrate of copper washed with water, first six times by decantation combined with filtration, and finally filtered through the filter paper and washed till free from copper. The filter and its contents are now spread out on a sheet of glass and the precipitate transferred by means of a platinum spatula to an 80 cc. beaker. Now add 5 cc. of E AgNO_3 solution, digest for half-an-hour (this to ensure perfect reaction), add 25 cc. of *standard* $\frac{\text{E}}{4} \text{H}_2\text{SO}_4$, allow to digest in an air oven for one hour at 50°C ., filter by decantation, and wash free from acid. The reaction which takes place here is as follows: $\text{Cu}(\text{NO}_3)_2\text{CuO} + \text{H}_2\text{SO}_4 = \text{Cu}_2(\text{NO}_3)_2 + \text{CuSO}_4 + \text{H}_2\text{O}$, so that if we ascertain how much acid the basic nitrate has taken up, we can calculate its equivalent of oxygen. In order to ascertain this, fill a burette with *standard* $\frac{\text{E}}{4} \text{Na}_2\text{CO}_3$, and run it gradually into the solution, which should be boiling (this is to eliminate CO_2 produced, which would keep any basic carbonate of Ag or Cu in solution) until a faint permanent precipitate is produced. Subtract the volume required from 25 cc. and this will give the amount of $\frac{\text{E}}{4} \text{H}_2\text{SO}_4$ neutralized.

Now 1 cc. of $\frac{\text{E}}{4} \text{H}_2\text{SO}_4 = .002$ grm. of oxygen, and in order

to get the percentage of oxygen present multiply the number of cc.'s of $\frac{E}{4}$ H_2SO_4 required by .002, divide by the amount of sample taken, and multiply by 100.

Estimation of Silver and Sulphur.—Take 200 cc. of stock solution = 10 grms. of sample, and dilute to 700 cc. with distilled water. Heat to $70^\circ C.$, add 2 cc. of 5 E HCl , allow the chloride of silver to settle, out of contact with light, for twenty-four hours, decant as much as possible of the clear fluid, filter off the $AgCl$ on a small filter paper (chemically pure), wash with $\frac{E}{5}$ HNO_3 and finally with water till free from copper salt. Dry in water oven, scrape off into a tared porcelain crucible, ignite filter separately, and add residue to main portion; moisten with a couple of drops of 16 E HNO_3 , to oxidize any reduced silver, add a drop of 10 E HCl to convert it into chloride, evaporate and carefully ignite at a dull red heat for ten minutes (do not fuse), cool in desiccator, and weigh. The weight of $AgCl$ thus ascertained, multiplied by $.7527 \times 100$ = percentage of silver present in sample.

The filtrate containing the sulphur is now evaporated to dryness with 50 cc. of 10 E HCl . The residue is taken up in 50 cc. of water, and evaporated to dryness again with 50 cc. of 10 E HCl (this is to convert the copper into chloride, as $BaSO_4$ is soluble to an appreciable extent in a strong solution of nitrate of copper). Dissolve the residue in 200 cc. of $\frac{E}{10}$ HCl , and filter if necessary; boil, add 10 cc. of E baric chloride solution, and allow to stand twenty-four hours; decant as much as possible of the clear solution, filter off the $BaSO_4$ on a small filter, wash with $\frac{E}{5}$ HCl , and finally with water until free from copper salts. Dry in water oven, brush off the precipitate into a tared crucible, ignite the filter separately, add residue to former, and ignite in oxidizing flame of Bunsen at a dull red heat for ten minutes. Allow to cool, and weigh.

The weight of BaSO_4 multiplied by $\cdot 1373 \times 10 =$ the percentage of sulphur present in the sample.

Estimation of Tin.—Weigh out 10 grms. of sample, brush into a tared litre flask with cork, add 96 cc. of 5 E HCl and 8 cc. 16 E HNO_3 ; heat until dissolved, dilute to 840 cc., add 160 cc. of 10 E potassic hydrate solution, saturate with SH_2 (this will take a couple of hours), allow the precipitate to subside, decant as much as possible of the clear supernatant liquor through a filter paper into another weighed litre flask, weigh and calculate for quantity of sample taken as in the example of estimation of Tin (*vide* page 37), remove from flask to a beaker, neutralize with 10 E HCl and add 30 cc. in excess. Allow the precipitate to subside in a warm place for a couple of hours; filter, wash with SH_2 water, and dry in water oven; brush the precipitate of tin, antimony, and arsenic sulphide with the free sulphur produced, into a tared Rose's crucible, provided with a lid and delivery tube; connect the delivery tube with the SH_2 apparatus, and pass current of dry SH_2 over; heat the crucible gently at first with a Bunsen flame, and gradually more strongly, so as to volatilize the free sulphur and arsenic sulphide. The sulphides of Sb and Sn now remain. Moisten with a couple of drops of 16 E HNO_3 , add 1 cc. of 22 E HNO_3 , allow to digest hot for half-an-hour, evaporate carefully, heat gradually, at first with lid on over Bunsen, and afterwards to red heat with lid off. Cool and weigh the SnO_2 and Sb_2O_4 thus produced, and subtract from this the weight of Sb_2O_4 as found in the other portion taken for the antimony estimation. Remainder = weight of SnO_2 . Multiply this by $\cdot 7867$, divide by amount of sample taken, multiply by 100 = percentage of tin in the sample.

[Test the residue for Cu, which, if present, must be extracted by means of nitric acid; and the residue again ignited and weighed.]

Estimation of Copper (*Brown's process*).—It sometimes happens that an estimation of the amount of copper present in

a sample of copper is sufficient in many cases. But in order to reduce the error involved in its estimation to a minimum great care is required and perfect confidence and experience in the method on the part of the operator to insure accuracy. The author has found the following modified process of Brown's to give very good results :—Dissolve .5 gm. of sample (weighed to $\frac{1}{10}$ of a milligram) in 10 cc. of 6 E HNO_3 , add 6 cc. 5 E H_2SO_4 , evaporate to dryness and heat residue on sand bath until fumes cease to come off; dissolve in 10 cc. of E H_2SO_4 , filter off sulphate of lead on a very small filter, wash with E H_2SO_4 until free from copper, dilute to 40 cc., gradually add an E solution of Na_2CO_3 until a slight permanent precipitate is produced in the cold, re-dissolve this in 1 cc. of 5 E H_2SO_4 and add 12 cc. of 2 E sodic acetate solution and 9 cc. of 5 E acetic acid; boil for ten minutes, filter off the basic acetate of iron, &c., without losing a drop of the solution, and wash with a hot $\frac{\text{E}}{10}$ solution of sodic acetate till free from copper salt. Reduce filtrate by evaporation to about 35 cc. and wash into a colourless 250 cc. stoppered bottle. Volume now 50 cc.; add to this 50 cc. of a solution of potassic iodide containing 5 grms. KI; shake well, and allow to stand for ten minutes. Now carefully run in from a burette an $\frac{\text{E}}{10}$ standard solution of hyposulphite of soda (thio-sulphate $\text{Na}_2\text{S}_2\text{O}_3$) until nearly all the free iodine liberated is seen to have disappeared; now add 5 cc. of starch solution (1 gm. starch in 100 cc. of water), which will form blue iodide of starch; run in the hyposulphite very carefully, stirring, until the solution becomes colourless. Note the volume of hyposulphite required. Taking the atomic weight of copper as 63.1, then 1 cc. of $\frac{\text{E}}{10}$ $\text{Na}_2\text{S}_2\text{O}_3$ should equal .00631 gm. copper; but the value of the hyposulphite should invariably be obtained by treating 0.5 gm. of pure electrotype copper as above and noting the volume of the $\frac{\text{E}}{10}$ hyposulphite that this is equivalent to. A

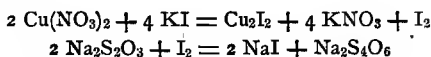
factor can thus be obtained for determining unknown quantities. Owing to a little difficulty that presents itself sometimes in determining the end of the reaction, due to differences in tint, a duplicate assay should go on at the same time, so that if the first assay was run a little over or a little under the mark, by a second observation this could be corrected. An example will make matters clearer.

·5 grm. of a sample of electrotype copper supplied by Messrs. Hopkin and Williams required 80 cc. of $\frac{E}{10}$ hyposulphite (theoretical quantity being 79·2 cc.) and a sample of tube-plate copper previously completely analyzed by the foregoing methods and giving 99·80 per cent. of copper by difference, required 79·8 cc. $\frac{E}{10}$ hyposulphite. Then

$$80 : 79·8 :: ·5 : x = ·49875$$

$$·49877 \times 2 \times 100 = 99·75 \text{ per cent. copper present.}$$

Reactions :



ANALYTICAL DATA OF A COMPLETE ANALYSIS OF A SAMPLE OF COPPER BY THE FOREGOING METHODS.

Residue insoluble in 6 E HNO₃.

50 grms. taken.

	Grms.
Watch glasses + clip + filter paper after drying in	
water oven for 2 hours	31·8997
Ditto + insoluble residue dried 1 hour in water oven .	38·9097
“ “ “ “ “ 2nd time	38·9097
<hr/>	
Insoluble residue in 50 grms.	·0100

$$·01 \times 2 = 0·02 \text{ per cent. insoluble residue.}$$

ESTIMATION OF IRON, NICKEL, &C.

Calculation for amount of sample taken.

Original sample taken = 10 grms.

	Grms.
Flask (1) + cork	131.92
Do. + liquid + CuS	1190.08
liquid + CuS	1058.16
CuS (corresponding to 10 grms. Cu)	15.07
Total liquid	1043.09
Flask (2) + cork	128.16
Ditto + filtered liquid	966.02
Liquid taken for analysis	837.86

$$1043.09 : 837.86 :: 10 : x = 8.032$$

Equivalent to 8.032 grms. of copper taken for the estimation of iron, nickel, &c.

IRON.

8.032 grms. of sample taken.

	Grms.
Porcelain Crucible marked x	7.4512
Do. + Fe ₂ O ₃ 1st ignition	7.4535
„ „ 2nd ignition	7.4535
Fe ₂ O ₃	0.023
Fe ₂ O ₃ into Fe7000
Fe00161

$$\frac{.00161}{8.032} = .00020$$

$$.0002 \times 100 = .02 \text{ per cent. iron.}$$

NICKEL.

8.032 grms. of sample taken.

	Grms.
Porcelain Crucible marked O	7.4514
Do. + NiO 1st ignition	7.4599
„ 2nd „	7.4599
NiO0085
NiO into Ni7867
	39335
	62936

$$\frac{.006687}{8.032} = .000832$$

$$.00668695$$

Equivalent to .083 per cent. nickel

ZINC.

8.032 grms. of sample taken.

Nil.

MANGANESE.

Nil.

ESTIMATION OF ARSENIC.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked 12	6.0715
Do. + $Mg_2As_2O_7$ 1st ignition	6.2364
„ 2nd ignition	6.2360
	<hr/>
Allowing for solubility of precipitate	.1645
	<hr/>
	.1677
$Mg_2As_2O_7$ into As4839
	<hr/>
	15093
	5031
	13416
	6708
	<hr/>
	.08115003

$$.08115 \times 10 = .812 \text{ per cent. arsenic.}$$

ESTIMATION OF ANTIMONY.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked 00	6.9265
Do. + Sb_2O_4 + F.A.	6.9276
„ 2nd ignition	6.9276
	<hr/>
Sb_2O_40011
Sb_2O_4 into Sb7922
	<hr/>
	.7922
	<hr/>
	.7922
	<hr/>
Sb.00087142

$$.000871 \times 10 = .009 \text{ per cent. antimony.}$$

ESTIMATION OF LEAD.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked <i>V</i>	6.9168
Do. + PbSO ₄ 1st ignition	6.9286
„ 2nd „	6.9286
PbSO ₄	0.0118
PbSO ₄ into Pb	6832
	<hr/>
	54656
	6832
	6832
	<hr/>
	0.00806176

$$0.008061 \times 10 = 0.081 \text{ per cent. lead.}$$

ESTIMATION OF BISMUTH.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked <i>u</i>	7.4906
Do. + Bi ₂ O ₃ 1st ignition	7.4959
„ 2nd „	7.4954
„ 3rd „	7.4954
Bi ₂ O ₃	0.0048
Bi ₂ O ₃ into Bi	8965
	<hr/>
	71720
	35860
	<hr/>
	0.00430320

$$0.0043032 \times 10 = 0.043 \text{ per cent. bismuth.}$$

ESTIMATION OF SILVER.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked <i>x y</i>	6.0715
Do. + AgCl 1st ignition	6.0724
„ 2nd „	6.0724
AgCl	0.0009
AgCl = Ag	7527
	<hr/>
	0.00067743

$$0.00067743 \times 10 = 0.007 \text{ per cent. of silver.}$$

ESTIMATION OF SULPHUR.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked <i>x y</i>	6·0720
Do. + BaSO ₄ 1st ignition	6·0761
„ 2nd „	6·0761
	<hr/>
	·0041
Deduct for sulphur in reagents	·0031
	<hr/>
	·0010

$$·001 \times ·1373 = ·0001373$$

$$·0001373 \times 10 = ·001 \text{ per cent. sulphur.}$$

ESTIMATION OF TIN.

Calculation of amount of sample taken.

Original sample taken = 10 grms.

	Grms.
Flask (1) + Cork	132·55
Do. + Liquid + CuS	1201·33
	<hr/>
Liquid + CuS	1068·78
CuS	15·07
	<hr/>
Total liquid	1053·71
Flask (2) + Cork	128·20
Do. + decanted liquid (filtered)	889·80
	<hr/>
Liquid taken for analysis	761·60

$$\text{Then } 1053·71 : 761·6 :: 10 : x = 7·228$$

Equivalent to 7·228 grms. sample taken for tin estimation.

	Grms.
Porcelain Crucible marked <i>T</i>	7·6287
Do. + SnO ₂ + Sb ₂ O ₄	7·6314
„ „ 2nd ignition	7·6314
	<hr/>
	·0027
Deduct Sb ₂ O ₄ (<i>vide</i> page 35) $\frac{·0011 \times 7·228}{10} =$	·0008
	<hr/>
SnO ₂	·0019

$$\frac{·0019 \times 7·867}{7·228} = ·000206 \text{ gm.}$$

Equivalent to ·021 per cent. tin.

ESTIMATION OF OXYGEN.

	Grms.
Watch glass	12.602
Do. + Sample	51.485
Sample taken	38.883
Weight of watch glass + sample after digestion in AgNO_3	46.559
Sample dissolved	4.926

The precipitated $\text{Ag} + \text{Cu}(\text{NO}_3)_2 \cdot \text{CuO}$, was digested with 5 cc. E AgNO_3 and 25 cc. *standard* $\frac{\text{E}}{4} \text{H}_2\text{SO}_4$, for one hour at 50°C ., filtered and washed. On titrating back with $\frac{\text{E}}{4} \text{Na}_2\text{CO}_3$ until a slight permanent precipitate was obtained on boiling, 23.9 cc. were required.

$$25 - 23.9 = 1.1 \text{ cc. } \frac{\text{E}}{4} \text{H}_2\text{SO}_4 \text{ used.}$$

$$1 \text{ cc. } \frac{\text{E}}{4} \text{H}_2\text{SO}_4 = .002 \text{ gm. O. or } .0178 \text{ gm. Cu}_2\text{O.}$$

$$1.1 \times .002 = .0022 \text{ gm. O.}$$

$$\frac{.0022}{4.926} = .000446 \text{ gm. O.}$$

$$.000446 \times 100 = .045 \text{ per cent. combined oxygen.}$$

$$1.1 \times .0178 = .01958 \text{ gm. Cu}_2\text{O}$$

$$\frac{.01958}{4.926} = .004$$

$$.004 \times 100 = .40 \text{ per cent. suboxide of copper, Cu}_2\text{O.}$$

ANALYSES OF TWO SAMPLES OF COPPER.

Nos. 1 and 2.

	No. 1. Per cent.	No. 2. Per cent.
Antimony	0.020	0.002
Arsenic	1.010	0.020
Bismuth	0.045	0.021
Iron	0.020	0.005
Lead	0.085	0.023
Nickel	0.083	0.069
Carried forward	1.263	0.140

		No. 1.	No. 2.
		Per cent.	Per cent.
Brought forward		1.263	0.140
Silver	. . .	0.010	0.014
Sulphur	. . .	Trace	Nil
Tin	. . .	"	0.018
Zinc	. . .	"	Nil
Oxygen	. . .	0.055	0.023
Copper (by difference)	. . .	98.672	99.805
Total		100.000	100.000

No. 1 is a sample of copper manufactured some forty years ago, and which has done good service as a locomotive fire-box. No. 2 is a sample of recent manufacture: it is a very good sample of copper for electrical purposes, &c., but owing to its purity and consequent softness, it did not last long as a locomotive fire-box.

The specification for copper tube plates for the Indian State Railways enacts that "the plates must be of the very best quality, and samples are to stand a test of having doubled cold without showing any signs of cracking. As many samples of the copper as the Inspector-General of the Rivay Stores shall think fit will be analysed by a metallurgist selected by the Inspector-General. Should any of the analyses show more than 0.5 per cent. in all of other metals or matters, the plates represented by that analysis will be rejected."

The writer's experience with regard to the quality of copper tube plates is that pure copper is too soft to be economically used; but what is the best hardening ingredient to add to copper so as to reduce corrosion and abrasion to a minimum, has yet to be determined; although arsenic in limited quantities would seem a favourable addition for that purpose.

IRON AND STEEL.

Methods for Complete Analysis.

Estimation of Silicon, Sulphur, Phosphorus, and Manganese.—Take 8 grms. of sample, brush into 500 cc. beaker, add 70 cc. of 16 E HNO_3 , heat gently until red fumes cease

to come off, add cautiously 20 cc. of 10 E HCl, boil until all that is soluble is dissolved, evaporate to complete dryness on hot plate, stirring, heat on sand bath for one hour at 200° C. to render silica insoluble, and to destroy organic matter, cool, add 60 cc. of 10 E HCl, and boil until all that is soluble goes into solution. Evaporate to 50 cc., dilute with distilled water to 120 cc., filter off silicon and graphite, remove any adhering to sides of beaker with the aid of a feather, and wash with hot 5 E HCl till free from Fe_2Cl_6 (this is ascertained by allowing some of the washings to drop into a test tube containing a little potassic sulphocyanide solution, when if iron is present a red coloration is produced). Reserve filtrate for the estimation of sulphur, phosphorus, and manganese. Dry the silica, &c., in the water oven, brush into a weighed crucible, ignite filter separately, add residue to main portion, ignite at a bright red heat for a quarter of an hour, cool and weigh the SiO_2 , and subtract filter ash from this weight. $\text{Remainder} \times \frac{4667 \times 100}{8}$

= percentage of silicon present.

This aqua regia method gives results for silicon slightly below the truth. Allen's method, herein described, is the most accurate, although both are frequently used, as is likewise the sulphuric acid method, which gives good results.

The hardening effect of silicon depends to a large extent upon the proportion of carbon present. As much as 0.5 per cent. of silicon in a steel, with only 0.1 per cent. of carbon, does not render the metal brittle, but if the carbon be as high as 0.5 per cent. with the silicon 0.5 per cent., the metal would be "red-short" and "cold-short." The addition of siliceous pig iron with a high percentage of manganese, to molten steel, makes sound castings, the silicon preventing the formation of blowholes owing to its deoxidizing any carbonic oxide present in solution before actual solidification. From a paper read by Mr. Turner before the Chemical Society, June, 1885, it seems that an appropriate addition of silicon to cast iron improves the tensile strength of the metal, as the following results that were obtained show:—

Silicon Per Cent.	Breaking Load. Tons per Square Inch.	Modulus or Elasticity.
0	10·14	25·79 millions.
0·5	12·31	28·67 „
1	12·72	31·18 „
2	15·70	23·56 „
2·5	14·62	25·45 „
3	12·23	21·15 „
4	11·28	15·64 „
5	10·16	18·72 „
7·5	5·34	14·75 „
10	4·75	13·93 „

Any further addition beyond 2·5 per cent. causes a gradual deterioration in strength.

Estimation of Sulphur.—The filtrate from the silica is now diluted to 160 cc. in a 200 cc. stoppered graduated test-mixer and thoroughly mixed, and 100 cc. of this (=5 grms. of sample), measured off into a 100 cc. graduated cylinder (the residual 60 cc. is reserved for the determination of phosphorus and manganese) and poured into a 200 cc. beaker. The free acid is neutralized as far as possible with 5 E AmHO (about 10 cc. will be found sufficient, which must be added gradually, stirring the solution thoroughly before each addition) heated to boiling, and 10 cc. of E BaCl₂ solution added, stirring. Allow to stand for twelve hours. Decant the supernatant fluid as far as possible from the precipitate, filter the residue through a small “pure” filter paper, wash until free from iron salts with $\frac{E}{10}$ HCl and finally with water, dry in water oven, brush BaSO₄ into a weighed porcelain crucible, ignite filter separately, and add the ash. Ignite the crucible and its contents for ten minutes at a dull red heat in an oxidizing flame, cool and weigh the BaSO₄, and multiply this weight by $\frac{1373 \times 100}{5}$.

Result = percentage of sulphur in sample.

Sulphur has a very deleterious effect upon iron, small quantities producing red-shortness. Manganese has a powerful influence in preventing the red-shortness of sulphur. Steel

containing 0.1 per cent. of sulphur and a low proportion of manganese is very liable to crack in rolling, while if the same proportion of sulphur be present, but with from 0.6 to 1 per cent. of manganese, such steel is known to roll without a flaw.

The following are analyses of steels by Herr Wasum containing various amounts of sulphur, with remarks on their rolling properties :—

No.	Carbon.	Silicon.	Phosphorus.	Manganese.	Sulphur.	Copper.	Rolling Qualities.
1	.280	.160	.049	.634	.119	.050	Good
2	.393	.141	.065	.695	.158	.040	Good
3	.258	.136	.043	.500	.201	.076	Bad
4	.307	.075	.039	.488	.214	.057	Bad
5	.224	.089	.030	.480	.231	.066	Very bad

Estimation of Phosphorus.—Measure off accurately 40 cc. (= 2 grms. of sample) of the original solution as left above (reserving the remaining 20 cc. for the estimation of manganese) into an 80 cc. beaker, add 6 cc. of 16 E HNO_3 , evaporate on water bath to dryness, redissolve in 6 cc. of 3 E HNO_3 , dilute to 15 cc., gradually add, stirring, 20 cc. of ammonic molybdate reagent (prepared by dissolving 55.5 grms. of molybdenum trioxide (MoO_3) in a mixture of 94 cc. of 20 E AmHO and 150 cc. of water and filtering into 694 cc. of HNO_3 sp.gr. 1.2 = 6.15 E, diluting to 1,000 cc. with 2 E HNO_3 , allowing to stand for twelve hours at 50° C. filtering and bottling), allow to stand for three hours at 40° C. with occasional stirring, filter through a small paper and wash until free from iron salts, first with $\frac{\text{E}}{10}$ HNO_3 (test the solution to ascertain if precipitation was complete by adding 10 cc. more of molybdate reagent to the filtrate and digesting at 40° C.), and then with water, until free from acid. Spread the filter on a watch glass and dry in water oven, brush off the yellow precipitate of phosphomolybdate of ammonium into a weighed crucible, and dry at 100° C. until weight remains constant. The precipitate as dried at

100° C. contains 1.63 per cent. of phosphorus, so that by multiplying by $\frac{.0163 \times 100}{2}$ we get percentage of phosphorus in sample.

Estimation of Manganese.—The residual 20 cc. of original solution (= 1 grm. of sample) left from above are poured into a litre flask, diluted to 250 cc., and neutralized with 5 E Am_2CO_3 solution, adding small quantities at a time, and vigorously shaking until a slight permanent precipitate is produced. This is now dissolved by adding two or three drops of 10 E HCl, and then adding 2 cc. in excess; 20 cc. of 5 E ammoniac acetate solution are now added, and the solution diluted to 800 cc., and boiled for ten minutes. Filter off quickly the basic acetate of iron $[\text{FeA}_3, \text{Fe}_2(\text{HO})_6]$ through a large ribbed filter paper into a 1,500 cc. flask, wash quickly half-a-dozen times with boiling distilled water, cool the filtrate to ordinary temperature, add 3 cc. of bromine, shake well until all is dissolved (the object of the bromine here is to convert the manganous acetate in solution into manganic acetate), allow to stand for five minutes and add 20 cc. of 20 E AmHO, boil for ten minutes, filter off the manganic hydrate $[\text{Mn}(\text{HO})_2]$ hot, wash with hot water until free from ammoniacal salts, dry in water oven, scrape precipitate into a tared crucible, ignite filter separately, add ash, gradually ignite to full red heat for five minutes, and then at a higher temperature over a blow lamp for five minutes, taking care to avoid reducing gases. Cool and weigh the Mn_3O_4 produced which $\times .7205 \times 100$ = percentage of manganese present in sample.

Estimation of Graphite and Silicon.—(*Allen's Method.*)—Weigh out 5 grms. of sample into a 200 cc. beaker, add 70 cc. of 5 E HCl, heat until all that is soluble is dissolved, filter into a 500 cc. beaker, wash the precipitate four or five times with $\frac{\text{E}}{10}$ HCl, and afterwards three or four times with water. The filtrate is now placed on a water bath to evapo-

rate. Wash the precipitate of silica and graphite into a silver crucible of about 50 cc. capacity, by means of a fine jet of water from a wash bottle, add to it 11 grms. of potassic hydrate, dilute to about 40 cc., put on cover and place in water bath for twenty minutes with occasional stirring. The silica is thus dissolved, the graphite being insoluble, which however always retains a small proportion of silica undissolved. Wash out into a beaker, dilute to about 120 cc. and filter off graphite, adding the filtrate to former on water bath, after previously adding 20 cc. of 10 E HCl. Evaporate to complete dryness and heat on sand bath to 160° C. for one hour, cool, moisten residue with 10 cc. of 10 E HCl, heat on water bath for half an hour, dilute to about 100 cc., boil, filter and wash with $\frac{E}{10}$ HCl till free from iron, and finally with distilled water till free from acid. Dry in water bath, brush into a weighed crucible, heat gradually to full redness for ten minutes, cool in desiccator, and weigh the SiO_2 .

$$\frac{\text{SiO}_2 \times .4667 \times 100}{5} = \text{percentage of silicon.}$$

The graphite on the filter is washed off into a small beaker by means of a jet of water from a wash bottle, and evaporated to dryness on a water bath, after which a mixture of 15 cc. 10 E HCl and 5 cc. 16 E HNO_3 is added, and boiled for ten minutes to dissolve out any iron; &c. Dilute to 60 cc., filter and wash till free from iron. Dry in water oven, brush precipitate into a weighed crucible, and reject filter paper. Heat in an air bath regulated to 160° C. for one hour, cool and weigh. Ignite until all graphite has burned off, cool and reweigh. The residue left here (should not exceed 2 milligrams) is deducted from the graphite, &c., as dried at 160° C., and the difference is taken as graphite, which $\times \frac{100}{5} =$ percentage graphite.

Estimation of Combined Carbon.—There are two methods in use—

(1) Combustion method, which involves the separation of the carbon, which is ignited in a current of oxygen, and the resulting CO_2 absorbed and weighed.

(2) Colorimetric method. In this method it is assumed that when a steel is dissolved in 6 E HNO_3 , the colour of the resulting solution is proportionate to the amount of combined carbon present. A steel in which the combined carbon has been estimated by the combustion method is taken as a standard, and the sample is diluted until the tints are coincident, when the percentage of carbon is easily deduced.

(1) *Combustion Method (Total Carbon).—Estimation of Combined Carbon and Graphite.*—Weigh out 5 grms. of the sample in a 300 cc. beaker, and pour on 250 cc. of Creath's solution. (This may be prepared in the following manner: Weigh out 170 grms. of pure recrystallized cupric chloride and 107 grms. of pure ammoniac chloride, and dissolve in 500 cc. of water, after which run in gradually from a burette $\frac{\text{E}}{10}$ NaHO

solution, stirring, until a permanent precipitate of cupric hydrate $[\text{Cu}(\text{HO})_2]$ is produced. Any free acid is thus neutralized. Filter off and dilute to 1,000 cc.) The solution is allowed to act upon the sample for about a quarter of an hour, with frequent stirring, and a gentle heat is applied until all the iron has gone into solution, which can be ascertained by pressing the precipitated copper with a glass rod, and observing if any particles of iron remain undissolved. When this is accomplished, the solution is heated to about 90°C . and kept at this temperature until all the copper has gone into solution, leaving the carbon, &c., suspended in the form of a fine precipitate. It now becomes necessary to filter off the carbon through an asbestos plug, preparatory to its combustion in oxygen. Some pure long-fibre asbestos is ignited at a bright red heat for about half an hour in a porcelain crucible, to destroy any organic matter present, and cooled in a place free from dust. A piece of tubing about an inch in diameter is drawn out as in A, Fig. 1, and a disc of perforated

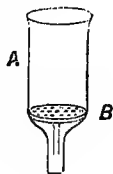


FIG. 1.

zinc, B, fitted into the contraction. Some of the asbestos is now roughly powdered in a clean mortar with a little water, and the whole poured on to the zinc plate, a quantity of asbestos being taken sufficient to make a stratum in the tube about an inch high. Another quantity of asbestos is

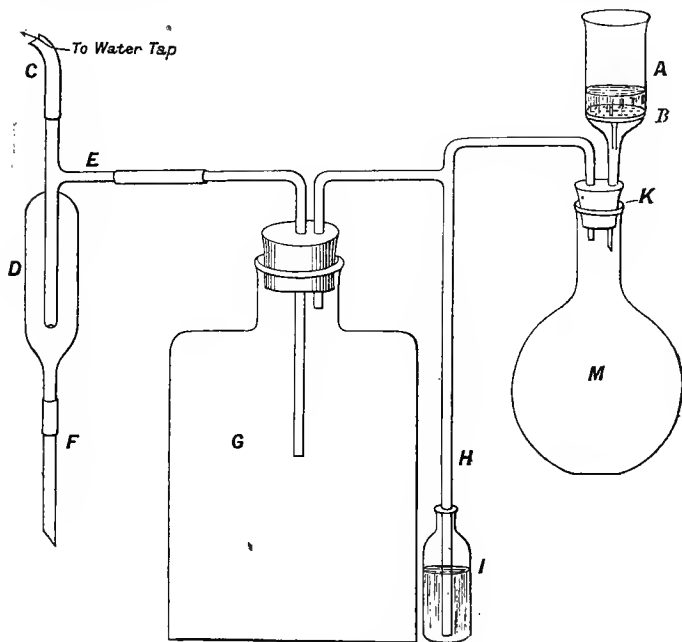


FIG. 2.

taken and powdered with water in the mortar very finely, and this is poured on the top of the other until a layer of about a quarter of an inch thick is obtained. This makes a good filter, capable of retaining the most finely divided precipitates. It is now fixed into the cork K of the flask M, Fig. 2, and filled with water, and a gentle stream of water made to pass through the ejector, D, when a partial vacuum is formed in M,

and the liquid is soon filtered into M. G is a reservoir, which serves to obtain a steady pull on A, the degree of vacuum being registered by the tube, H, which dips into mercury contained in the bottle, I. The solution containing the precipitated carbon is now filtered through A, with the aid of the ejector (about 3" of vacuum will usually be found sufficient), and washed with 5 E HCl till free from copper chloride, and the acid afterwards washed out with distilled water, and allowed to drain as far as possible. A is taken



FIG. 3.

out and put into a water oven to dry, B having been previously pushed half-way up the tube; when perfectly dry, the disc and its contents are pushed out of the tube with a glass rod on to a clean watch glass, and the under portion of the asbestos which does not contain any admixed carbon is detached as far as possible by means of a pair of forceps. The remainder containing the whole of the carbon is then placed into a platinum boat (Fig. 3), which is afterwards introduced into the combustion tube, D, in position shown in Fig. 4. As will

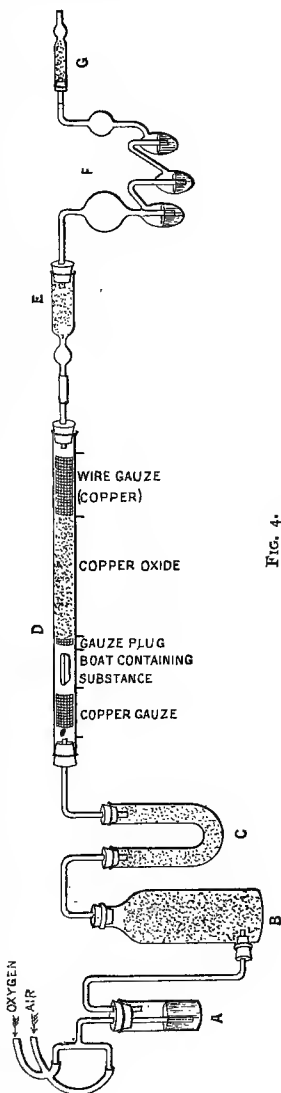


FIG. 4.

be seen in the sketch, the tube, besides the boat, contains copper gauze and copper oxide in the proportions seen; the tube should be about 28 in. long and about $\frac{1}{2}$ in. in diameter. The copper oxide should be in a granular form and recently ignited. The cylinder, A, contains 36 E H_2SO_4 , and is connected with the tubes x and y to gas holders containing oxygen and air respectively, and also to the cylinder, B, which is filled with good recently ignited soda lime; c is a U tube containing small pieces of caustic potash. These three tubes serve to absorb any moisture or CO_2 that is present in the oxygen or air that passes through the tube; E is a tube containing CaCl_2 , which absorbs any moisture that may be given off from the CuO , &c.; F is Giessler's potash bulbs which contain 8 E KHO solution, and G is a small tube containing CaCl_2 , which serves to absorb any moisture given off from F. When all is ready for combustion, F and G are accurately weighed and attached to E, the tube, D, of course being placed in a combustion furnace. The ends of the tube containing the copper gauze are first gradually heated to redness, a gentle current of oxygen being made to pass through the apparatus. The copper oxide is then gradually heated to its full length to bright redness, and then the part containing the boat is likewise gradually heated to bright redness, the oxygen passing through the potash bulbs about the rate of a bubble a second; after going for about an hour, all the carbon being oxidized, the oxygen is turned off and air turned on at about the same rate; meanwhile the gas is turned out, and after the air has been passing through for about a quarter of an hour, F and G are detached and weighed.

Increase in weight $\times \frac{27273 \times 100}{5} =$ percentage of total carbon.

Determination of Graphite by combustion. — 10 grms. of the sample of steel, or 2 grms. of pig iron are dissolved in 60 cc. of 8 E HCl , and diluted to 120 cc., and the graphite, &c., filtered off in the manner as described for total carbon. When all iron and acid are washed out, some warm E NaHO solution is allowed to percolate through; this is washed out with water

and the graphite dried and estimated by the above process. In deducting the graphite thus found from the total carbon, the amount of combined carbon is hereby attained.

The following is an example of an actual determination of graphite and combined carbon in a steel rail by the foregoing process.

TOTAL CARBON.

Five grms. of sample taken.

Potash Bulbs before combustion	Grms.
„ „ after „	37·7658
„ „ after „	37·8444
Wt. of CO ₂	·0786

$$\frac{·0786 \times ·27273 \times 100}{5} = ·429 \text{ per cent. total carbon.}$$

GRAPHITE.

Ten grms. of sample taken.

Potash Bulbs before combustion	Grms.
„ „ after „	38·0313
„ „ after „	38·0434
Wt. of CO ₂	·0121

$$\frac{·0121 \times ·27273 \times 100}{10} = ·033 \text{ per cent. graphite.}$$

COMBINED CARBON.

$$·429 - ·033 = ·396 \text{ per cent. combined carbon.}$$

(2) *Estimation of Combined Carbon by Eggertz' Colorimetric Method.*—The principle of this method, as already stated, is, that when steel is dissolved in 6 E HNO₃ a more or less brown colour is produced, which may be taken as being in proportion to the amount of combined carbon present. A standard steel is first selected, the combined carbon in which has been previously determined by combustion. It is important that the amount of carbon present in the standard should be somewhat near to that expected in the sample. It would be wise to keep at least three standards

(1) containing about 1 per cent. carbon for tool steels, &c.; (2) containing .40 per cent. for rails, tyres, &c.; (3) containing .20 per cent. for axles and soft steels generally. Suppose we have to determine the combined carbon in a sample of rail steel by this method. Select two test tubes, 5 in. by $\frac{1}{2}$ in., and weigh into them 0.1 grm. of the 0.4 per cent. standard,



FIG. 5.

and 0.1 grm. of the sample respectively, and add to each 2 cc. of 6 E HNO_3 . A beaker of water or a copper bath arranged with a perforated false bottom, through which the test tubes can pass, is made to boil, and the test tubes put in and allowed to stay in the boiling water for exactly a quarter of an hour with occasional shaking, after which they are taken out and allowed to cool. The standard is now poured out into a graduated tube (Fig. 5), holding 20 cc. graduated in $\frac{1}{16}$ cc., and diluted to 8 cc., each cc. of which will now equal .05 per cent. of combined carbon. Pour the sample into a similar tube, and dilute with small quantities of water delivered from a wash bottle, and compare tints after each addition, until the colours are seen to be as near as possible alike. A piece of tissue paper held behind the tubes renders the observation more exact. Suppose that when 6.5 cc. was reached the colours were alike, the amount of carbon present would be $6.5 \times .05 = .325$ per cent.

It appears that 1.5 per cent. of carbon in a steel is the maximum amount that can be present to render it workable. Steel with 1 per cent. of carbon welds well, and makes good cold chisels.

The following are ideal percentages of carbon in various steels, assuming the other elements being present in normal quantities.

Per cent.	
1.3 to 1.5	Razors and Chilled Roll Tools.
1.2 to 1.4	Saw Files.
1.1 to 1.2	Drills, Turning Tools, &c.

Per cent.

- 1 to 1·1 Spindles, large Turning Tools, Millpicks, &c.
 ·8 to 1·0 Shears, Dies, Cold Sets, &c.
 ·3 to ·5 Tyres, Rails, Cannon, &c.
 ·2 to ·3 Boiler Plates, &c.
 ·15 to ·20 Crank Axles, Tinplate Iron, &c.

The hardness of a steel containing a given quantity of carbon will depend upon the rate at which it was cooled, and also upon the amounts and nature of other elements that may be present, such as silicon, phosphorus, manganese, &c. The following analyses show that excess of silicon in presence of excess of carbon is injurious to steel used for tyres, axles, and steel plates for bridges.

	BAD STEEL.		GOOD STEEL.	
	1 Per cent.	2 Per cent.	1 Per cent.	2 Per cent.
Combined Carbon and Graphite	·40	·53	·35	·49
Silicon	·59	·64	·05	·01
Phosphorus	·01	·03	nil	·03
Sulphur	·01	·06	„	·03
Manganese	nil	·05	„	·57
Copper	nil	·03	·01	·02

The following interesting results were obtained by Mr. Spencer, showing the influence of hardening and annealing upon the condition of carbon in steel.

No. 1 SAMPLE.	Before Hardening. Per cent.	Hardened. Per cent.	Annealed. Per cent.
Combined Carbon (colour test)	·0·89	0·58	0·98
Graphitic Carbon . . .	0·29	trace	0·20
Total Carbon	1·18	0·58	1·18
Total Carbon by combustion	1·18	1·09	1·20

Loss 0·51

No. 2 SAMPLE.	Before Hardening. Per cent.	Hardened. Per cent.	Annealed. Per cent.
Combined Carbon (colour test)	0·80	0·66	0·79
Graphitic Carbon . . .	0·34	0·07	0·44
Total Carbon	1·14	0·73	1·23
Total Carbon by combustion	1·21	1·10	1·19

Loss ·37

No. 3 SAMPLE.	Before Hardening. Per cent.	Hardened. Per cent.	Annealed. Per cent.
Combined Carbon (colour test)	0.38	0.22	0.38
Graphitic Carbon . . .	0.04	nil	trace
Total Carbon	0.42	0.22	0.38
Total Carbon by combustion	0.39	0.34	0.38

Loss . . . 0.12

Sulphuric Acid Method for the Estimation of Silicon.

—The following method will be found to give good results for silicon determinations:—10 grms. of the sample are weighed out into a 400 cc. beaker, dissolved in 120 cc. of 5 E H_2SO_4 , evaporated as far as possible on a water bath, and then heated on a sand bath or hot plate until fumes of H_2SO_4 are evolved. The beaker is now cooled, 120 cc. of distilled water poured cautiously on to the residue, the solution boiled up for a short time, the insoluble silica filtered off, washed till free from acid, and determined as directed on page 44.

Estimation of Copper.—Weigh 1 grm. of the sample into a 100 cc. beaker, add 12 cc. of 6 E H_2SO_4 , heat until all iron has gone into solution, dilute to 50 cc. and boil; now add gradually a solution of 3 E thiosulphate of soda ($\text{Na}_2\text{S}_2\text{O}_3$), as long as a black precipitate continues to form, and on allowing to settle the supernatant liquid contains suspended sulphur only; (from 1 to 2 cc. of thiosulphate of soda are usually required) the copper is thus precipitated as subsulphide (Cu_2S). Filter off through a small filter and wash until free from iron with distilled water, dry in a water oven, carefully ignite filter and precipitate in a good porcelain crucible, digest with 2 cc. of 10 E HCl + 1 cc. of 16 E HNO_3 until dissolved, evaporate nearly to dryness, add $\frac{1}{2}$ cc. of water, make slightly alkaline with 5 E AmHO , filter off any iron, &c., and receive the filtrate in a carbon tube (Fig. 5), washing the filter a little with 2 E AmHO in small quantities until the blue tint thus produced is the same as that of a standard copper solution contained in a similar tube, and which is prepared by dissolving 0.1 grm. of pure electrotype copper in 1 cc. of 6 E HNO_3 and 2 cc. of 10 E

HCl, and diluting to 500 cc. with 2 E AmHO at 15.5° C., 1 cc. thus containing .0002 grm. of copper. Supposing that the volume of the sample was 5 cc. when the tints were coincident, $5 \times .0002 \times 100 = 0.10$ per cent. of copper present.

The effect of copper on steel was generally considered to render it red-short, but Herr Wasum has shown that as much as 0.86 per cent. of copper may be present, which yet rolls perfectly sound. The following are analyses and the effects of copper, and of copper and sulphur, on the rolling properties of steel obtained by Herr Wasum.

INFLUENCE OF COPPER.

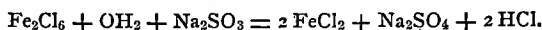
	Carbon.	Silicon.	Phos- phorus.	Manga- nese.	Sulphur.	Copper.	Rolled.
No. 1	.276	.144	.064	.778	.059	.452	Very Good.
No. 2	.233	.091	.050	.709	.060	.862	Good.

INFLUENCE OF COPPER AND SULPHUR.

	Carbon.	Silicon.	Phos- phorus.	Manga- nese.	Sulphur.	Copper.	Rolled.
No. 1	.311	.051	.061	.514	.107	.859	Good.
„ 2	.281	.169	.059	.594	.170	.429	Bad.
„ 3	.235	.164	.045	.468	.173	.573	Bad.
„ 4	.262	.131	.052	.655	.189	.406	Bad.

Estimation of Iron.—Usually in iron and steel analyses the iron is taken by difference after estimating all the other elements present. It sometimes happens, however, that a direct determination of iron is required, when the following is the best method to adopt. Weigh out .56 grm. of the sample into a small assay flask of about 80 cc. capacity, add 10 cc. of 10 E HCl, and heat until dissolved; then add in small portions at a time about $\frac{1}{2}$ grm. of potassic chlorate, to oxidize organic matter, &c., and evaporate down to about 4 cc. Transfer to a bulb flask of about 500 cc. capacity, supplied with a rubber cork into which a tube with a Bunsen valve is fitted. Dilute to 200 cc., then add 10 cc. of 2 E Na_2SO_3 , and its equivalent of HCl, viz. 2 cc. of 5 E. Attach cork and tube, and boil until free from SO_2 , which usually takes about a quarter of

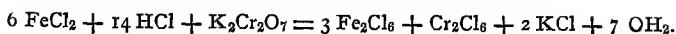
an hour or twenty minutes after it starts boiling. Close the valve quickly and put the flask into cold water to cool. The iron is thus reduced to the form of ferrous chloride, as the following reaction shows:—



The next step in the process is to ascertain how much of an oxidizing agent is necessary to convert the FeCl_2 into Fe_2Cl_6 . In this case bichromate of potash ($\text{K}_2\text{Cr}_2\text{O}_7$) is the most suitable to use.

When the solution is cold, pour it into a beaker of about 500 cc. capacity, and test, to ascertain if perfectly reduced; by bringing a drop of the solution on the end of a glass rod in contact with a drop of E potassic sulphocyanide solution on a porcelain plate; when if a mere trace of Fe_2Cl_6 is present a red coloration will be produced. If no coloration is produced add 10 cc. of 10 E HCl. Fill a 100 cc. burette graduated in 0.1 cc. with *standard* $\frac{\text{E}}{10}$ potassic bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), prepared by dissolving 4.917 grms. of the pure, dry, recrystallized salt in about 500 cc. of pure distilled water, and diluting to 1000 cc. at 15.5°C . (This solution should be standardized by the method here described, taking .56 gm. of pure pianoforte wire, when 99.7 cc. of the $\frac{\text{E}}{10}$ solution should be required, the 0.3 cc. being allowed for unavoidable impurities in the iron). Run the $\frac{\text{E}}{10} \text{K}_2\text{Cr}_2\text{O}_7$ into the iron solution at the rate of about $\frac{1}{10}$ cc. per second, with continual stirring, bringing a drop of the solution out from time to time on the end of a glass rod and mixing with a drop of dilute solution (.5 gm. in 500 cc.) of potassic ferricyanide, when a more or less blue coloration is produced as long as there is any ferrous chloride present. When no further blue or green colour is produced with the ferricyanide on allowing to stand for four minutes, the reaction may be considered completed. Note the number of cc.'s required.

The following is the action of the bichromate :—



Supposing that 98·7 cc. were required, then, since ·56 gm. of the sample was taken and 1 cc. of $\frac{\text{E}}{10} \text{ K}_2\text{Cr}_2\text{O}_7 = \cdot0056 \text{ gm. Fe}$, the amount of iron present is 98·7 per cent.

In addition to the determination of the element just described in iron and steel, there are, of course, special steels in which tungsten, chromium, aluminium, or titanium may be present. If the character of the iron or steel is unknown, it would be safer to make first a qualitative analysis of the sample, and the result thus obtained would decide what elements are to be determined. The following are methods for the estimation of the above-named four elements :—

Estimation of Aluminium.—The following is the process adopted by Mr. Stead for estimating small quantities of aluminium in iron and steel :—Weigh out 11 grms. of the sample in a 600 cc. beaker, add 44 cc. of 10 E HCl, heat until dissolved, and evaporate to dryness. Redissolve residue in 60 cc. of E HCl, filter off into a 500 cc. beaker, and wash the residue till free from iron. Dilute the filtrate to 150 cc., add 3 cc. of E sodic phosphate solution, and gradually add 5 E AmHO until the free acid is neutralized and a permanent precipitate is produced. Now add 10 E HCl drop by drop until the solution has cleared up, heat to boiling and add 50 cc. of a saturated solution of sodic thiosulphate, and continue boiling for one hour; filter and wash. Dissolve the precipitate off the filter with 10 cc. of hot 5 E HCl, collecting the filtrate in a platinum dish, wash the filter and evaporate to dryness, add 2 grms. of sodic hydrate and 1 cc. of water; allow to dissolve, evaporate to dryness, and fuse for about ten minutes over a Bunsen. Cool and digest with 50 cc. of water, and dilute to exactly 110 cc.; filter off the insoluble oxides and measure off 100 cc. of the filtrate (= 10 grms. original sample), neutralize with 10 E HCl, add 3 cc. of E sodic phosphate and 30 cc. of the

saturated thiosulphate solution, and boil; now add 2 cc. of 5 E ammoniac acetate, and boil five minutes longer and filter; wash the precipitate with hot water, dry, ignite, and weigh the AlPO_4 . $\text{AlPO}_4 \times .2236 \times 10 = \text{percentage of aluminium.}$

Estimation of Chromium.—(*Arnold's Method.*)—Weigh out 2 grms. of the sample into a 100 cc. beaker, add 20 cc. of 10 E HCl, and heat until dissolved; evaporate over the water bath to complete dryness; detach as much as possible of the crisp residue with the aid of a glass rod, and brush on to a clean watch-glass; any chloride still clinging to the beaker is dissolved off with a few cc. of 5 E HCl, and poured into a deep platinum crucible and evaporated to complete dryness. The main portion of the chlorides on the watch glass is now brushed into the crucible and the whole finely powdered with a glass rod; 5 grms. of fusion mixture (1 part Na_2CO_3 and 1 part KNO_3) are now introduced and thoroughly mixed with the chlorides. The mixture is now fused for about fifteen minutes over a blow-lamp. The iron is thus converted into insoluble oxide, and the chromium, silicon, and manganese into alkaline chromate, silicate, and manganate respectively. Allow to cool, put the crucible and contents into a beaker containing about 80 cc. of boiling water, and digest until all that is soluble is dissolved; take out the crucible with the aid of a glass rod, and wash the liquid adhering to it into the beaker with hot water. Now add 1 cc. of alcohol to the solution to decompose the manganate, and stir well and allow the oxides of iron and manganese to thoroughly settle. Decant the clear liquid through a double filter (without disturbing the precipitate) into a beaker; add 30 cc. of boiling water to the residue, stir, allow to settle, drain off the clear liquid through the filter as before, wash the filter twice with hot water, and treat the residue with a second 30 cc. of hot water and filter as before; this may be considered sufficient washing for the oxides. The solution, which now contains chromate and silicate, is acidified with 20 cc. of 10 E HCl and boiled until all CO_2 and nitrous fumes have come off. Now add gradually 5 E AmHO until slightly

alkaline, heat nearly to boiling, and filter off the resulting precipitate through an ashless filter and wash. Dissolve the precipitate off the paper with 10 cc. of hot 5 E HCl, wash the paper into the solution and evaporate to complete dryness on a sand bath to render the silica insoluble; dissolve residue in 100 cc. of E HCl, filter off silica, collecting filtrate into a porcelain basin, neutralize the solution with 20 E AmHO, adding about $\frac{1}{2}$ cc. in excess and heat to incipient boiling, filter off the chromic hydrate $\text{Cr}_2(\text{HO}_6)$ through an ashless filter, wash with boiling water till free from soluble matter, dry in water oven, and ignite carefully in weighed crucible for ten minutes at a good red heat. Cool, and weigh the resulting Cr_2O_3 .

$$\frac{\text{Cr}_2\text{O}_3 \times .6862 \times 100}{2} = \text{per cent. chromium.}$$

An appropriate addition of chromium to steel increases its tensile strength. Chrome steel is manufactured in Brooklyn, containing from 1 to 2 per cent. of chromium, by reducing chrome-iron ore with charcoal in crucibles, and mixing the crude chromium thus produced with a suitable quantity of wrought iron.

Mr. Arnold has made experiments with steel tyres containing chromium, and the result shows that this element materially increases the tensile strength of the tyres over ordinary Bessemer steel. The following are results that he obtained.

The composition of an ordinary steel tyre was:—

	Per cent.
Carbon	0.280
Silicon	0.070
Manganese	1.250
Sulphur	0.080
Phosphorus	0.080

The mechanical tests of the above would give a maximum tensile strain of 37 tons per square inch; elongation, 26 per cent.; reduction of area, 47 per cent.; and fracture, grey

granular with silky edges, shape convex and concave. Such a tyre, with an inside diameter of 2 ft. 8 in. and a sectional area of 11 in., would behave under the falling weight test in a manner indicated by the subjoined figures, the weight of the tup being 22 cwt.:—

Fall in Feet	.	.	2'	4'	6'	8'	10'	12'	} unbroken.
Deflection in Inches	.	.	$\frac{1}{2}$ "	$\frac{3}{4}$ "	$1\frac{3}{4}$ "	$3\frac{1}{16}$ "	$4\frac{1}{16}$ "	$6\frac{1}{2}$ "	

Steel was made, having the following composition:—

	Per cent.
Carbon	. 0.25
Silicon	. 0.03
Manganese	. 1.75
Sulphur	. 0.12
Phosphorus	. 0.11

The mechanical tests gave a mean maximum strain of 42.1 tons per square inch; mean elongation, 18 per cent.; mean reduction of area, 26.3 per cent. The mean results of the tests on many tyres, practically identical in composition, 2 ft. 8 in. inside diameter, and having a sectional area of 11 in., the weight of the tup being 22 cwt., were:—

Fall in Feet	.	.	2'	4'	6'	8'	10'	12'	14'
Deflection in Inches	.	.	$\frac{1}{8}$ "	$\frac{5}{8}$ "	$1\frac{3}{8}$ "	$2\frac{3}{16}$ "	$3\frac{9}{16}$ "	$5\frac{1}{8}$ "	$6\frac{1}{4}$ "

The analysis of a tyre steel required to stand a strain of at least 48 tons per square inch was:—

	Per cent.
Carbon	. 0.28
Chromium	. 0.42
Manganese	. 1.54
Silicon	. 0.08
Sulphur	. 0.10
Phosphorus	. 0.09

The mechanical tests gave, maximum tensile strain, 49.8 tons per square inch; elongation, 15 per cent.; reduction of area, 26 per cent.; fracture flat and finely crystalline. A tyre of

2 ft. 8 in. diameter and of 11 in. sectional area behaved thus under the 22 cwt. falling weight:—

Fall in Feet	2'	4'	6'	8'	10'	12'	14'	16'	18'	20'	25'
Deflection in Inches	$\frac{1}{8}$ "	$\frac{1}{2}$ "	$1\frac{1}{8}$ "	$1\frac{1}{2}$ "	$3\frac{1}{16}$ "	$4\frac{3}{8}$ "	$5\frac{7}{8}$ "	$7\frac{5}{8}$ "	$9\frac{1}{2}$ "	$11\frac{3}{8}$ "	broke.

A test piece planed out of the broken tyre gave the following results:—Maximum tensile strain, 47·7 tons per square inch; elongation, 3 per cent.; reduction of area, 6·4; fracture, large crystals. The molecular change set up by the shock and vibration of the falling weight is thus clearly indicated.

Estimation of Titanium.—(*Riley's Method*).—Weigh out 10 grms. of the sample into a 500 cc. beaker, pour on to it 60 cc. of 16 E HNO_3 , and heat on a hot plate until its action on the iron has nearly ceased; now add cautiously 20 cc. of 10 E HCl , boil until all that is soluble is dissolved, evaporate to dryness, heat the residue on a sand bath to about 130°C ., for an hour, cool, add 50 cc. of 10 E HCl , and boil until all that is soluble is dissolved. The solution, which should now be about 40 cc., is diluted to 80 cc., and the graphite and silica, with some titanic acid, are filtered off and washed with hot $\frac{\text{E}}{10} \text{HCl}$ till free from iron. The filtrate is reserved, the residue is dried and ignited in a platinum crucible, then fused with about 16 times its weight of bisulphate of potash, and then cooled and digested with hot water until all that is soluble is dissolved; it is now filtered, and the filtrate added to the reserved filtrate containing the iron, &c. Pour the mixed filtrates into an 800 cc. flask, provided with a cork fitted with a Bunsen valve, dilute to 300 cc., add 100 cc. of 2 E Na_2SO_3 and 20 cc. of 10 E HCl , and boil until the iron solution is reduced and all sulphurous odour has gone off. Cool, add 1 cc. of E HNO_3 , nearly neutralize with ammonia, add 80 cc. of 5 E ammoniac acetate, boil for a few minutes, and filter off the precipitate quickly, which contains, besides titanic acid, oxide of iron and phosphate of iron. Wash

with $\frac{E}{10}$ acetic acid, dry, ignite in platinum crucible, and fuse with 16 times its weight of bisulphate of potash; digest in cold water, and filter off phosphate of iron, &c. Dilute the filtrate to 500 cc., add 1 cc. of 16 E HNO_3 , and boil for about six hours, keeping up the original volume with additions of boiling water. The titanous acid is now allowed to settle over night, the clear liquid decanted, the residue filtered, washed, dried, and ignited in a platinum crucible, and then cooled and weighed. The weight of TiO_2 thus produced $\times .6098 \times 10 =$ percentage of titanium.

Estimation of Tungsten.—Weigh out 5 grms. of the sample, dissolve in 120 cc. of 6 E HNO_3 , evaporate to dryness on sand bath, cool, add 40 cc. of 10 E HCl , and boil until all that is soluble is dissolved; add 40 cc. of water and again boil, filter off the precipitate, which may consist of oxide of tungsten (WO_3), graphite, Fe_2O_3 , TiO_2 , &c., wash with hot water, dry and ignite in a platinum crucible; add 10 cc. of hydrofluoric acid (HF) and 2 cc. of 36 E H_2SO_4 , evaporate in a hood, and repeat the evaporation with HF and H_2SO_4 until all SiO_2 has escaped as SiF_4 ; add to the residue four times its weight of Na_2CO_3 , fuse over a Bunsen for ten minutes, cool, and treat the fused mass with water until all that is soluble is dissolved.

Filter off insoluble oxides, &c., and wash with $\frac{E}{5}$ Na_2CO_3 .

The solution now contains all the tungsten as soluble tungstate of soda. Dilute to 100 cc., neutralize with 16 E HNO_3 , boil off CO_2 , and add two drops of 16 E HNO_3 in excess; add 50 cc. of $\frac{E}{5}$ mercurous nitrate, neutralize the free acid by adding sufficient recently precipitated oxide of mercury, and stir well. Allow to settle, filter off the mercurous tungstate with the slight excess of oxide of mercury, and wash with hot water till free from soda salts; dry in water oven, scrape into platinum crucible, ignite filter separately, and add ash. Now heat the crucible under a hood gradually to a bright red heat, until fumes

have all come off. The mercury thus completely volatilizes, and leaves the tungsten in the form of oxide (WO_3). Cool and weigh.

$$\frac{\text{WO}_3 \times .7931 \times 100}{5} = \text{percentage tungsten.}$$

ANALYTICAL DATA OF AN ACTUAL ANALYSIS, BY THE ABOVE METHODS, OF A BAD SAMPLE OF BESSEMER STEEL.

ESTIMATION OF COMBINED CARBON.

(*Eggertz' method.*)

0.1 gm. of sample taken.

The total volume of solution of sample, when the tint was equal to the standard solution (half strength) was 8.6 cc.

Now each cc. of the *dilute standard* solution contains .00005 gm. of carbon, therefore,

$$\frac{8.6 \times .00005 \times 100}{0.1} = 0.43 \text{ per cent. combined carbon.}$$

ESTIMATION OF SILICON.

(*Sulphuric acid method.*)

10 grms. taken.

	grms.
Porcelain Crucible marked 6	7.5183
Do. + SiO_2 + F. Ash	7.5842
" " " 2nd ignition	7.5840
	<hr/>
	.0657
F.A.0005
	<hr/>
$\text{SiO}_2 =$0652
SiO_2 into Si4667
	<hr/>
	4564
	3912
	3912
	2608
	<hr/>
	.03042884

$$\frac{.0304 \times 100}{10} = 0.304 \text{ per cent. of silicon.}$$

ESTIMATION OF SULPHUR.

5 grms. taken.		grms.
Porcelain Crucible marked 7	7.3390
Do. + BaSO ₄ + F.A. + BaSO ₄ from reagents used	7.3633
" " " " 2nd ignition	7.3631
		<hr/>
BaSO ₄ + F.A. + Re0241
F.A. + Re0019
		<hr/>
BaSO ₄0222
BaSO ₄ into S1373
		<hr/>
		666
		1554
		666
		222
		<hr/>
		.00304806

$$\frac{.003048 \times 100}{5} = 0.061 \text{ per cent. of sulphur.}$$

ESTIMATION OF PHOSPHORUS.

2 grms. taken.		grms.
Porcelain Crucible marked x	3.7960
Do. + "ppt" dried at 100° C	3.8242
" " " 2nd drying	3.8242
		<hr/>
		.0282
"ppt" into P0163
		<hr/>
		846
		1692
		282
		<hr/>
		.00045966

$$\frac{.0004596 \times 100}{2} = 0.023 \text{ per cent. of phosphorus.}$$

ESTIMATION OF MANGANESE.

(Bromine method.)

1 gm. taken.

	grms.
Porcelain Crucible marked Q	7.3386
Do. + Mn ₃ O ₄ + F. Ash.	7.3535
" -- " " 2nd ignition	7.3534
Mn ₃ O ₄ + F.A.0148
F.A.0017
Mn ₃ O ₄0131
Mn ₃ O ₄ into Mn7205
	655
	262
	917
	.00943855

$$.009438 \times 100 = 0.944 \text{ per cent. of manganese.}$$

ESTIMATION OF COPPER.

(Colour method.)

1 gm. taken.

Volume of blue solution when tint was equal to that of the standard = 3.75 cc.

$$1 \text{ cc. of Standard Solution} = .0002 \text{ gm. Cu.}$$

therefore,

$$3.75 \times .0002 \times 100 = 0.075 \text{ per cent. of copper.}$$

ANALYSES OF VARIOUS IRONS AND STEELS.

Analysis of a Sample of Virginian Meteoric Iron (Mallet).

Iron	88.706
Nickel	10.163
Cobalt	0.396
Copper	0.003
Tin	0.002
Manganese	trace
Phosphorus	0.341
Sulphur	0.019
Chlorine	0.003
Carbon	0.172
Silica _{SiO₂}	0.067
	99.872

ANALYSES OF PIG IRONS.

	North- ampton. Hot Blast.	Hematite. Bessemer Pig No. 1.	West Hallum. No. 3 Foundry.	Kirkless Hall. No. 3.
Graphite . . .	1·150	3·050	2·650	3·200
C. Carbon . . .	·554	·710	—	—
Silicon . . .	1·900	2·012	1·350	1·275
Sulphur . . .	0·414	0·010	0·055	0·081
Phosphorus . . .	1·807	0·052	0·650	1·750
Manganese . . .	0·395	0·040	0·415	2·215
Iron (by difference)	93·780	94·126	94·880	91·479
	100·000	100·000	100·000	100·000

ANALYSES OF VARIOUS CAST IRONS.

	Gurlt.			Bodemann.		Abel.	
	Grey Coal.	Mottled Hot Blast.	White Gart- sherrie.	Grey Hot Blast.	Mottled Cold Blast.	Grey. French Charcoal.	White Silesian.
Density .	7·21	7·21	7·41	7·166	7·43	7·000	7·531
Carbon com- bined .	1·021	1·793	2·457	1·44	2·78	—	4·94
Graphite .	2·641	1·110	0·871	2·71	1·99	3·40	—
Silicon .	3·061	2·165	1·124	3·21	0·71	0·80	0·75
Sulphur .	1·139	1·480	2·516	trace	trace	0·05	trace
Phosphorus	0·928	1·171	0·913	1·22	1·23	0·45	0·12
Iron .	90·236	89·314	89·863	91·42	93·29	95·18	88·57
Manganese.	·834	1·595	2·715	trace	trace	—	5·38
Copper .	—	—	—	—	—	—	0·24
	99·860	98·629	100·459	100·00	100·00	99·88	100·00

ANALYSIS OF A BAD SAMPLE OF BESSEMER STEEL RAIL BROKEN
BY WEIGHT OF LOCOMOTIVE.

Combined Carbon	0·275
Silicon	0·235
Sulphur	0·037
Phosphorus	0·065
Manganese	0·750
Iron (by difference)	98·638
	100·000

The high percentage of silicon in this rail was no doubt the cause of breaking.

ANALYSIS OF A BESSEMER STEEL RAIL THAT GOT BROKEN DURING
TRANSFERENCE FROM RAIL-BANK TO WAGGON.

Combined Carbon	.	.	.	0.350
Silicon	.	.	.	trace
Sulphur	.	.	.	0.080
Phosphorus	.	.	.	0.230
Manganese	.	.	.	0.150
Iron (by difference)	.	.	.	99.190
				<hr/>
				100.000

The high percentage of phosphorus, making the steel "cold short," was no doubt the cause of breakage. Often chemical analysis does not show up any reason why a rail should break. In these cases it is either due to a flaw, or to a want of physical uniformity due often to careless manufacture.

The author considers that an ideal rail and tyre should have the following compositions, provided that a thorough homogeneous metal is insured upon:—

	Rail.	Tyre.
Combined Carbon	0.350 to 0.400	0.400 to 0.500
Silicon	0.056 ,, 0.070	0.080 ,, 0.100
Sulphur	0.040 ,, 0.060	0.040 ,, 0.060
Phosphorus	0.040 ,, 0.060	0.040 ,, 0.060
Manganese	0.750 ,, 1.000	0.400 ,, 0.600

ANALYSIS OF A KRUPP TYRE THAT HAS DONE GOOD SERVICE.

Combined Carbon	.	.	.	0.500
Silicon	.	.	.	0.275
Sulphur	.	.	.	0.035
Phosphorus	.	.	.	0.060
Manganese	.	.	.	0.475
Iron (by difference)	.	.	.	98.655
				<hr/>
				100.000

It will be observed that this tyre contains a high percentage of silicon, and although it wore well it would be unsafe to

generally recommend such steel for the purpose, especially if manufactured by the Bessemer process. With 0.5 per cent. of carbon in a tyre the amount of silicon should not exceed 0.1 per cent.

The following results show that the elements are not evenly distributed in an ingot (Snelus) :—

	Top of Ingot.	Bottom of Ingot.
Combined Carbon	0.760	0.350
Silicon	trace	trace
Sulphur	0.187	0.044
Phosphorus	0.191	0.044
Manganese	0.558	0.514

Six samples were taken out of the top of an ingot and six out of the bottom from positions sketched in section (Fig. 6), when the following results were obtained :—

	FROM SECTION AT TOP.			FROM SECTION AT BOTTOM.		
	Carbon.	Sulphur.	Phosphorus.	Carbon.	Sulphur.	Phosphorus.
1	.44	.032	.044	.44	.048	.060
2	.54	.048	.066	.42	.056	.062
3	.57	.080	.086	.41	.048	.054
4	.61	.096	.097	.40	.048	.054
5	.68	.120	.111	.38	.048	.058
6	.77	.187	.142	.37	.044	.052

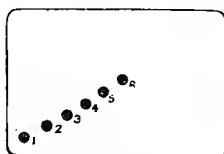


FIG. 6.

ANALYSIS OF STEEL USED FOR DIES IN ROYAL MINT.

	(1)	(2)	(3)	(4)
Carbon (combined)	0.82	1.07	0.79	1.19
Manganese	0.10	0.12	0.24	0.45
Silicon	0.05	0.06	0.18	0.29
Sulphur	trace	trace	0.01	trace
Phosphorus	—	„	0.01	„

ANALYSIS OF SWEDISH GUN-BARREL STEEL.

Carbon	0.450
Silicon	0.450
Manganese	0.400
Sulphur	0.040
Phosphorus	0.085
Iron (by difference)	98.575
						<hr/> 100.000

ANALYSIS OF TWO SAMPLES OF KRUPP-GUN STEEL OF APPROVED QUALITY.

	(1)	(2)
Combined Carbon	.42	.69
Graphite	.04	.02
Silicon	.11	.06
Manganese	.13	.15
Phosphorus	nil	trace
Sulphur	—	—
Copper	.30	.26

SPECIFICATION OF QUALITY OF STEEL SUPPLIED TO THE COCKERHILL WORKS, SERAING, FOR THE PURPOSE OF SHIPBUILDING.

Carbon08 to .15
Silicon	trace „ .02
Sulphur03 „ .05
Phosphorus03 „ .05
Manganese30 „ .60

SPIEGELEISEN AND FERROMANGANESE.

Estimation of Manganese.—There are two methods generally in use, (1) Direct method, and (2) Indirect method.

Direct method.—Weigh out .5 gm. of the finely-powdered sample into a 50 cc. assay flask, add a mixture of 10 cc. 10 E HCl and 5 cc. 22 E HNO₃ (fuming), and boil until all action ceases. Now add a few crystals of potassic chlorate to ensure complete oxidation of organic matter, and boil until all

free chlorine is eliminated, and bulk of liquid is reduced to about 8 cc. ; pour into a $1\frac{1}{2}$ litre flask, wash out with water, dilute to 250 cc., and gradually add 5 E ammonic carbonate solution, until a slight permanent precipitate is obtained which does not disappear on vigorously shaking. Dissolve the precipitate in two or three drops of 10 E HCl and add 2 cc. in excess. Now add 20 cc. of 5 E ammonic acetate solution, dilute to 800 cc., boil for ten minutes, filter off quickly the basic acetate of iron thus produced, and wash three times with hot distilled water. The precipitate is dissolved off the paper with 5 cc. 10 E HCl into a $1\frac{1}{2}$ litre flask, diluted to 250 cc., and iron precipitated and filtered off as before. The two filtrates are now mixed in a large beaker, and evaporated down to 1 litre, allowed to cool, 3 cc. of bromine added, and stirred until all goes into solution. The rest of the process is conducted as stated on page 43.

Indirect method.--Estimate the percentage of iron volumetrically by the process described on page 53, add to this result 5 per cent. for carbon and impurities, and the difference between this and 100 is taken as the percentage of manganese present. This indirect method is largely used in Spiegel works, &c. ; and where a number of estimations are required in one day, it may be considered sufficiently accurate for all practical purposes. In cases of dispute between buyers and sellers the direct method must be adopted. Fresenius gives the following analysis of Spiegeleisen made from Stahberg spathic ore.

	Per cent.
Iron	82.860
Manganese	10.707
Nickel	0.016
Cobalt	trace
Copper	0.066
Aluminium	0.077
Titanium	0.006
Magnesium	0.045
Carried over	93.777

	Brought forward	Per cent.
	.	93.777
Calcium	.	0.091
Potassium	.	0.063
Arsenic	.	0.007
Antimony	.	0.004
Phosphorus	.	0.059
Sulphur	.	0.014
Nitrogen	.	0.014
Silicon	.	0.997
Carbon	.	4.323
Slag	.	0.665
		<hr/> 100.014

Manganese can be made to replace iron in pig iron to the extent of 85 per cent. When pig iron contains manganese from about 5 to 20 per cent. it is called spiegeleisen, and when over this amount ferromanganese. Manganese plays an important part in the manufacture of steel. Owing to its greater affinity for oxygen than iron it deoxidises any FeO formed in the process; the resulting MnO forming a fluid slag with the impurities readily separating from the molten metal. It likewise neutralises to a great extent the deleterious effects of sulphur.

BRASS, BRONZE, &c.

Estimation of Tin, Lead, Copper, Iron and Zinc.—The following process is applicable to the analysis of Aich's metal, bell metal, brass (all colours), bronze for bearings, Dutch metal, gun metal, Muntz metal and sterro metal. Although all the above-named elements are not always present in these alloys, it would be always advisable to pass them through the following process. If it is thought that the amount of any one element present, judging from the amount of precipitate found, is not sufficient in quantity to affect the quality of the alloy, of course it could be filtered off and discarded, and the estimation of the more important metals proceeded with. Weigh out 1 grm. of the sample into an 80 cc.

low and wide beaker, add 15 cc. of 6 E HNO_3 , and heat until all that is soluble is dissolved. If any quantity of tin be present a white precipitate of an hydrate of tin, called metastannic acid, is produced. Evaporate on the water bath nearly to dryness, dilute with distilled water to 100 cc., boil, filter off the precipitate, wash with $\frac{1}{10}$ E HNO_3 , until the filtrate gives no reaction for copper with potassic ferrocyanide, and then with distilled water until free from acid. The filter and its contents are spread out on a watch glass and put into a water oven to dry; in the meantime a small porcelain crucible is weighed, after which the precipitate is brushed into it, the filter ignited separately, and the ash added to main portion, finally ignited for ten minutes over the blow-lamp, and weighed. The weight of the oxide of tin (SnO_2), thus produced $\times .7867 \times 100 =$ percentage of tin present. The filtrate, which may now contain lead, copper, iron and zinc as nitrates, is evaporated as low as possible on the water bath, with addition of 2 cc. of 36 E H_2SO_4 , and finally on the sand bath until nearly all fumes of H_2SO_4 are driven off. Digest residue with 50 cc. $\frac{\text{E}}{2} \text{H}_2\text{SO}_4$ till all that is soluble is dissolved, filter off PbSO_4 , wash with $\frac{\text{E}}{2} \text{H}_2\text{SO}_4$ until free from copper, and finally with water until free from acid. Dry in water oven, scrape precipitate into weighed porcelain crucible, ignite filter separately, add ash to main portion, ignite at a dull red-heat for ten minutes, and weigh. Weight of $\text{PbSO}_4 \times .6832 \times 100 =$ percentage of lead. The filtrate is mixed with 23 cc. 10 E HCl , and diluted to 350 cc., heated to 70°C ., saturated with sulphuretted hydrogen, and the precipitated copper sulphide allowed to subside, and then filtered off and washed quickly with SH_2 water. To ensure the perfect separation of zinc, the precipitate is dried, roasted carefully in a porcelain capsule, dissolved in 10 cc. of aqua regia (3 cc. 16 E HNO_3 and 7 cc. 10 E HCl), evaporated to dryness, residue dissolved in 52 cc. of 5 E HCl , and diluted to 350 cc., and the copper separated as sulphide as before; the two filtrates are mixed and reserved

for the determination of zinc and iron. The CuS is dissolved off the filter with 10 cc. of 8 E HNO_3 , using small portions at a time, and collected in a 300 cc. porcelain basin; the filter is washed free from Cu , the solution diluted to 200 cc., and heated nearly to boiling; 2 E NaHO solution is now run in, until no further precipitation of copper hydrate takes place, and the solution shows a slight alkaline reaction. It is now boiled, allowed to settle, and the clear liquid decanted as far as possible through a filter paper; 150 cc. of hot water are poured on to the residue and again boiled up, the whole filtered off, and the precipitate washed with hot water till free from alkali. The filter and its contents are now dried in water oven, the CuO scraped into a weighed crucible, the filter paper ignited separately, the ash added to main portion, and the whole ignited for ten minutes at a dull red heat over a Bunsen. The weight of the CuO thus obtained $\times .7985 \times 100 =$ percentage of copper.

The reserved filtrates containing any iron and zinc are evaporated to 200 cc., with addition of a few crystals of KClO_3 for oxidation, poured into a 300 cc. porcelain dish, heated nearly to boiling, and 2 E Na_2CO_3 run in gradually, stirring until all the iron and zinc present are precipitated as basic carbonates, and the solution shows a distinct alkaline reaction. It is now boiled up, allowed to settle, the clear liquid filtered, the residue washed three times with boiling water by decantation combined with filtration, the precipitate finally washed on the paper until free from alkali, dried in the water oven, scraped into a weighed platinum crucible, the filter paper ignited in usual manner, and ash added to main portion. The crucible and its contents are now heated to bright redness for ten minutes, cooled, and weighed. The increase in weight = $\text{ZnO} + \text{Fe}_2\text{O}_3$. Dissolve the mixed oxides in about 5 cc. of 10 E HCl , wash out into a 250 cc. bulb flask, reduce with 2 E Na_2SO_3 , and estimate the amount of $\frac{\text{E}}{10} \text{K}_2\text{Cr}_2\text{O}_7$ taken up by the reduced iron on titration by the process given on page 53. The number of cc.'s required $\times .0056 \times 100 =$

percentage of iron present in the sample. To ascertain the percentage of zinc let

$$\text{ZnO} + \text{Fe}_2\text{O}_3 = \text{X}.$$

$$\text{Fe}_2\text{O}_3 = \text{Y}.$$

$$\text{ZnO} = \text{Z}.$$

$$\text{Y} = \text{cc. } \frac{\text{E}}{10} \text{ K}_2\text{Cr}_2\text{O}_7 \text{ required} \times .008.$$

$$\text{Z} = \text{X} - \text{Y}$$

and $\text{Z} \times .8026 \times 100 = \text{percentage of zinc}.$

The following is an example of analytical data of a bad sample of brass, by the foregoing process :—

ESTIMATION OF LEAD.

1 grm. of sample taken.

Porcelain Crucible marked W	7.7159
Do. + PbSO ₄	7.7336
„ „ 2nd ignition	7.7336
						<hr/>
						PbSO ₄ .0177

$$.0177 \times .6832 = .01209264.$$

$$.01209264 \times 100 = 1.21 \text{ per cent. Pb.}$$

ESTIMATION OF COPPER.

Porcelain Crucible marked V	8.2226
Do. + CuO + F.Ash.	8.8626
„ „ „ 2nd ignition	8.8623
„ „ „ 3rd „	8.8623
						<hr/>
						.6397
						F.A. .0029
						<hr/>
						.6368

$$.6368 \times .7985 \times 100 = 50.85 \text{ per cent. Cu.}$$

ESTIMATION OF ZINC.

Small Platinum Crucible	13.8316
Do. + ZnO + Fe ₂ O ₃ + F.Ash	14.4401
" " " " 2nd ignition	14.4385
" " " " 3rd "	14.4385
	<hr/>
	.6069
F.A.	.0029
	<hr/>
(X) ZnO + Fe ₂ O ₃	.6040
(Y) Fe ₂ O ₃ (see below)	.0496
	<hr/>
(Z) ZnO	.5544

$$.5544 \times .8026 \times 100 = 44.49 \text{ per cent. Zn.}$$

ESTIMATION OF IRON.

The .6042 grm. ZnO + Fe₂O₃ was dissolved in 10 E HCl, and the Fe estimated therein by standard $\frac{E}{OI}$ K₂Cr₂O₇ in the usual manner, when 6.2 cc. were required for complete oxidation.

$$1 \text{ cc. of } \frac{E}{IO} \text{ K}_2\text{Cr}_2\text{O}_7 = .0056 \text{ grm. Fe.}$$

$$1 \text{ cc. of } \frac{E}{IO} \text{ K}_2\text{Cr}_2\text{O}_7 = .008 \text{ " Fe}_2\text{O}_3$$

$$6.2 \times .008 = .0496 \text{ grm. Fe}_2\text{O}_3.$$

$$6.2 \times .0056 \times 100 = 3.47 \text{ per cent. Fe.}$$

SUMMARY.

	Per cent.
Lead	1.21
Iron	3.47
Copper	50.84
Zinc	44.49
Tin	traces
	<hr/>
	100.01

When a sample of an alloy is submitted for analysis, and its nature unknown, it would perhaps be wise to first make a careful qualitative analysis, which may in some cases modify the *modus operandi* of the quantitative analysis.

The following table gives analyses of various alloys, which can be analysed by the process just described.

	Tin.	Copper.	Iron.	Zinc.
Aich's Metal	—	60	1·8	38·2
Bell Metal	22	78	—	—
Brass, Best	—	71·4	—	28·6
„ Common	—	66·6	—	33·4
„ Yellow	—	60	—	40
„ Pin Wire	—	63	—	37
Bronze Coinage . . .	4	95	—	1
„ for Bearings . . .	16	82	—	2
„ „ „	17	6	—	77
„ „ Wheel Boxes . .	18	80	—	2
Dutch Metal	—	84·6	—	15·4
Sterro Metal	1·5	58	3·0	37·5
Speculum Metal . . .	33·4	66·6	—	—
Muntz Metal	—	60	—	40
Stopcocks and Pump Valves	10	88	—	2

WHITE METALS.

The following alloys, which may contain tin, antimony, lead, copper, iron or aluminium, such as Britannia metal, Babbit's metal, solder, type metal, and pewter (triple and ley), may be analysed by the process here described. Weigh out 1 grm. of sample in fine shavings or drillings into an 80 cc. low and wide beaker, add 14 cc. 8 E HNO_3 , heat nearly to boiling on a hot plate until all that is soluble is dissolved, evaporate nearly to dryness on water bath, add 50 cc. of distilled water to the residue, boil until all that is soluble is dissolved, allow to settle, pour off the clear liquid through a filter, add 20 cc. hot water to the residue, boil up, and filter off as before. Continue this treatment three times, and finally filter off the precipitate which may be metastannic acid and antimonious acid, and wash with hot water until the washings scarcely redden litmus paper; reserve the filtrate (No. 1), dry the precipitate in the water oven, brush into a silver crucible of about 30 cc. capacity, ignite filter paper, and add ash. Now add to the precipitate 8 times its bulk of pure sodic hydrate (note the

weight added), fuse over a Bunsen for a quarter of an hour, treat the fused mass with water containing 33 per cent. by volume of alcohol until all that is soluble is dissolved, transfer to a beaker and dilute with the alcohol solution until the strength of the solution becomes E with the quantity of sodic hydrate taken. It is now allowed to stand with frequent stirring for twelve hours. The stannate of soda and excess of sodic hydrate are now in solution, while the antimoniate of soda is precipitated. Filter off and wash with alcohol (30 per cent.), and finally with strong alcohol. Reserve filtrate (No. 2), dissolve the precipitate off the filter with 25 cc. 2 E HCl, in which there are previously dissolved 4 grms. of tartaric acid,

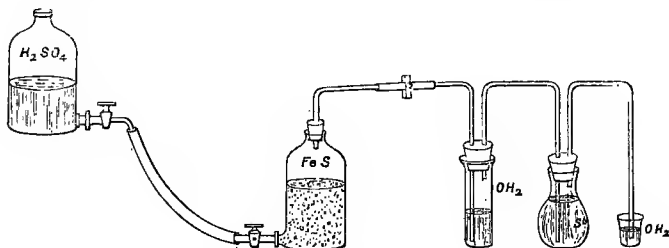


FIG. 7.

and collect in small flask. Dilute to 100 cc., fit cork into flask into which are fitted two tubes, one bent at right angles and going to the bottom of the flask, and the other bent twice at right angles, and just passing through the cork. Connect with SH_2 apparatus and dip the off-tube bent at right angles into a small beaker of distilled water. Fig. 7 speaks for itself with regard to the fittings.

Pass SH_2 until saturated, applying heat gently at first and finally to the boil. After complete precipitation has been insured, allow the flask to stand on a warm plate for half an hour and then pass CO_2 through until the odour of SH_2 is eliminated. Filter quickly, wash with SH_2 water, dry in water oven, scrape off the antimonious sulphide into a small beaker, ignite the filter, and add ash. Moisten with 16 E HNO_3 ,

gradually add 5 cc. of 22 E HNO_3 , heat on plate until oxidation is complete, pour into a weighed porcelain crucible, rinsing out the last portions of precipitate with distilled water with the aid of a wash bottle, evaporate to dryness, and gradually ignite to full red heat. Cool and weigh the Sb_2O_3 thus produced, which $\times .7922 \times 100 =$ percentage of antimony.

The filtrate (No. 2) containing the tin is evaporated until all alcohol is expelled. Neutralize with 10 E HCl , add 5 cc. in excess, dilute to 100 cc., saturate with SH_2 , allow to stand in a warm place until SH_2 is nearly all driven off, filter, and wash with a solution containing 5 per cent. 5 E ammoniac acetate and 5 per cent. 5 E acetic acid, till free from chlorides. Dry in water oven, scrape precipitate into a watch glass, ignite filter in a weighed crucible, oxidize ash with a drop of 22 E HNO_3 , and ignite. Now add main portion from watch glass, place lid on crucible, heat gently at first, then take lid off and increase the temperature gradually until SO_2 comes off freely; when this abates, heat strongly over the blow-lamp for ten minutes, cool, add a small lump of ammoniac carbonate, and ignite first over Bunsen with lid on till Am_2CO_3 has all gone off, and finally over the blow-lamp as before. Repeat until no further loss occurs. Cool and weigh. The weight of the SnO_2 thus produced $\times .7867 \times 100 =$ percentage of tin present.

To the filtrate (No. 1) containing the lead, copper, &c., add 10 cc. of 10 E H_2SO_4 , evaporate to dryness, heat up on sand-bath till all H_2SO_4 fumes have gone off, digest with 30 cc. of E H_2SO_4 until all that is soluble is dissolved; filter off PbSO_4 , wash with E H_2SO_4 till free from any copper, remove filtrate, wash the acid out of filter with strong alcohol, dry, and proceed as directed on page 70. Evaporate the filtrate to dryness, dissolve residue in 50 cc. E HCl , saturate with SH_2 , digest, filter off CuS , washing quickly with SH_2 water, treat and estimate as directed on page 71. The solution, which may contain small quantities of iron and aluminium, is boiled to expel SH_2 , and evaporated to about 30 cc., with addition of a few crystals of KClO_3 to convert FeCl_2 into Fe_2Cl_6 . The solution is now made slightly alkaline with 20 E AmHO , 5

cc. 5 E AmCl added, and boiled. If any appreciable quantity of ferric or aluminic hydrate is here precipitated, it is to be filtered off, and the iron and aluminium determined therein as directed on page 89. SH_2 may be passed through the ammoniacal filtrate, and any Zn, Ni, Mn, or Co will be precipitated if present, and may be determined by the methods given in the analysis of copper.

The following is an example of analytical data obtained in the analysis of a sample of Babbit's metal by the above process:—

ESTIMATION OF TIN.

1 grm. of sample taken.

Porcelain Crucible marked L.	7.1427
Do. + SnO_2 + F.A.	7.9273
„ „ „ 2nd ignition	7.9274
	<hr/>
	.7847
F.A.0029
	<hr/>
	.7818

$$.7818 \times .7867 \times 100 = 61.50 \text{ per cent. tin.}$$

ESTIMATION OF ANTIMONY.

Porcelain crucible marked C	7.0916
Do. + Sb_2O_3 + F.A.	7.2267
„ „ „ 2nd ignition	7.2264
	<hr/>
	.1348
F.A.0017
	<hr/>
	.1331

$$.1331 \times .7922 \times 100 = 10.54 \text{ per cent. antimony.}$$

ESTIMATION OF LEAD.

Porcelain crucible marked Y	7.8115
Do. + PbSO_4 + F.A.	8.2005
„ „ „ 2nd ignition	8.2003
	<hr/>
PbSO_4 + F.A.3888
F.A.0010
	<hr/>
	.3878

$$.3878 \times .6832 \times 100 = 26.49 \text{ per cent. lead.}$$

ESTIMATION OF COPPER.

Porcelain crucible marked F	6.7891
Do. + CuO + F.A.	6.8082
" " " 2nd ignition	6.8082
<hr/>	
Cu + F.A.0191
F.A.0010
<hr/>	
CuO0181

$$.0181 \times .7985 \times 100 = 1.45 \text{ per cent. copper.}$$

SUMMARY.

	Per cent.
Tin	61.50
Lead	26.49
Antimony	10.54
Copper	1.45
Iron, Zinc, Aluminium, &c.	traces
<hr/>	
	99.98

ANALYSES OF A FEW WHITE ALLOYS.

	Antimony.	Copper.	Tin.	Lead.	Zinc.
Britannia Metal .	6.2	1.8	92	—	—
Pewter, Triple .	15.0	—	79	6	—
" Ley .	—	—	80	20	—
Solder, Fine .	—	—	66.6	33.4	—
" Common .	—	—	50	50	—
" Coarse .	—	—	33.4	66.6	—
" Brazing .	—	50	—	—	50
Type Metal .	25	—	—	75	—
" " .	25	—	25	50	—

TIN PLATE.

Estimation of Tin.—It sometimes happens, that a knowledge of the thickness of tin on a tin plate is required. The following method has been proved to give good results:—Weigh out 5 grms. of the sample in chips into an 80 cc. bulb flask provided with a cork, into which passes a tube with a

Bunsen valve attached, add 53 cc. of 5 E HCl, dissolve (the object of dissolving the sample out of contact with the air, is to prevent precipitation of a large amount of sulphur by subsequent treatment with SH_2 , if the iron were present as perchloride), pour into a 300 cc. beaker, dilute to 250 cc., add 13 cc. of 10 E HCl, saturate with SH_2 , allow to stand in warm place for half an hour, filter off SnS , and wash with SH_2 water. Dissolve the SnS off the paper with 12 cc. of hot 2 E Na_2S , in which 0.1 grm. of sulphur is previously dissolved, by frequent percolation, and repeat with 12 cc. more. The solutions are now mixed, diluted to 100 cc., neutralized with 10 E HCl, adding 5 cc. in excess, allowed to stand in warm place for an hour, and the stannic sulphide filtered off and treated as described on page 31, in order to separate any arsenic sulphide that may be present, when the tin may be determined as oxide. A sample of tin plate gave by this process 2.5 per cent. of tin.

ANALYSIS OF WHITE LEAD.

Pure white lead is supposed to be a mixture of carbonate and hydrate of lead, but often it is mixed with, and sometimes wholly substituted by, such substances as chalk, heavy spar (barium sulphate), gypsum, witherite (baric carbonate), zinc oxide, kaolin, &c. For paints it is usually previously mixed with oil and "dryers," a compound which may contain anything in the shape of matter, often of doubtful efficiency.

Before making a quantitative analysis of a sample, it is well to first subject it to a careful qualitative analysis, and if not pure, to determine the nature and quantity of the adulterants.

Qualitative Analysis.—Weigh out about a grm. of the sample, add 20 cc. of 8 E HNO_3 , heat on hot plate until all that is soluble is dissolved, suck up any oil floating on the surface of the liquid with a piece of blotting or filter paper, dilute to about 40 cc., filter, and wash with $\frac{\text{E}}{10}$

HNO_3 . If any residue is present it may consist of BaSO_4 , PbSO_4 , SiO_2 , &c.; pour on about 10 cc. of hot 5 E ammoniac acetate, pouring it through several times. Any PbSO_4 is thus dissolved out, which will give a yellow precipitate of PbCrO_4 , when a solution of E K_2CrO_4 is added to it. The residue is fused with four times its weight of potassic carbonate and tested in the usual manner for acids and bases. The original nitric acid solution is diluted to about 150 cc., and conducted through the usual groups of reagents. If there is no insoluble residue on treatment with 8 E HNO_3 , and if, after the lead has been separated by SH_2 a little of the solution leaves no residue on evaporation on platinum foil, it may be taken for granted that there would not be much present in the way of the usual adulterants.

Method for Quantitative Analysis.—We will assume that the qualitative analysis shows the following to be present:—Oil, BaSO_4 , BaCO_3 , CaCO_3 , CaSO_4 , PbCO_3 and $\text{Pb}(\text{HO})_2$. Weigh out 2 grms. of the sample into a two-ounce stoppered bottle, pour on 20 cc. of methylated ether, tie down stopper, heat the bottle and its contents to about 40°C ., thoroughly shake, and allow the sediment to subside. When cool, withdraw stopper and decant the clear ethereal solution of the oil through a double filter into a weighed flask. Repeat the digestion with ether three times, finally filtering off the residue, and washing with ether until free from oil.

Distil off the ether by putting the flask, attached to a Liebig's condenser, into a beaker of hot water, and weigh the residual oil. The residue on the filter is dried and brushed into a flask, and the CO_2 determined therein by the method described on page 87, using 8 E HNO_3 for the decomposition instead of HCl . The solution from this determination is now filtered, and the residue, which will consist of BaSO_4 and CaSO_4 , is washed with $\frac{\text{E}}{10} \text{HNO}_3$. Reserve solution. The residue is dried, brushed into an 80 cc. porcelain basin, and 20 cc. of 2 E Na_2CO_3 poured on to it and boiled for about an hour, keeping up the original bulk with hot water; the

CaSO_4 is thus completely decomposed, insoluble calcic carbonate being formed, together with soluble sulphate of soda. The solution is diluted to 50 cc., and the CaCO_3 and BaSO_4 filtered off. Since the CaCO_3 on the filter is the equivalent of the CaSO_4 originally present, the solution may be rejected, the CaSO_4 being deduced from the amount of CaO found hereafter. Percolate 20 cc. of hot E HCl through the precipitate several times, wash the BaSO_4 remaining on the filter, dry, determine in usual manner. Test residue for PbSO_4 . Dilute the solution to about 30 cc., add 5 cc. 5 E AmCl , 5 cc. of 20 E AmHO , and 10 cc. of E ammonic oxalate; stir well, and allow the calcic oxalate to settle for a few hours in a warm place, after which it is to be filtered off, washed, dried, ignited gradually to a bright red heat, cooled, and the resulting CaO weighed. Every 7 parts of CaO equal 17 parts of CaSO_4 .

The reserved nitric acid solution containing lead, &c., is now evaporated nearly to dryness, 2 cc. of 10 E HCl added, the solution diluted to 150 cc. with hot water, saturated with SH_2 , the PbS allowed to subside, filtered off, washed with SH_2 water, dissolved off the filter in 8 E HNO_3 and precipitated and determined as sulphate as directed on page 70. The filtrate is made alkaline with 10 cc. of 20 E AmHO , 5 cc. of 5 E Am_2S added, and then allowed to stand a short time in a warm place. Any precipitate that may be formed is filtered off and examined for zinc, iron, &c. The filtrate contains the barium and calcium originally present in the sample, as carbonates. Add to the solution 10 cc. of 5 E Am_2CO_3 , allow to stand some time, and filter off the carbonate of Ca and Ba through a weighed filter. The solution might be tested for magnesia by adding to it a solution of sodic phosphate, and allowing to stand twelve hours. It now becomes necessary to determine the proportion of CaCO_3 and BaCO_3 present in the precipitate; this may be done indirectly, since there is such a wide difference in the atomic weights of Ba and Ca . Dry the precipitate and weigh. Determine the CO_2 present by the method employed for the estimation of total CO_2 , the difference between these two amounts being the quantity combined as carbonate of lead. To

calculate the amounts of BaCO_3 and CaCO_3 from the data obtained—

Let $X = \text{weight of } \text{BaCO}_3 + \text{CaCO}_3.$

$Y = \text{CO}_2 \text{ found.}$

$Z = \text{CaCO}_3 \text{ in mixture.}$

then

$\text{CO}_2 \quad \text{EqBaCO}_3$

$44 : 197.2 : : Y : C \text{ (BaCO}_3, \text{ if all present as such).}$

$C - X = D$ which is proportional to amount of CaCO_3 present.

$\text{BaCO}_3 - \text{CaCO}_3 \quad \text{CaCO}_3$

$97.2 : 100 : : D : = Z \text{ (CaCO}_3)$

and $X - Z = \text{BaCO}_3.$

PART II.

ORES, LIMESTONES, ETC.

IRON ORES.

Method for Complete Analysis.—The ore, having been carefully sampled, is quartered and powdered moderately fine, taking care that the last portion completely passes through the smallest sieve. It is thoroughly mixed and placed in a dry stoppered bottle.

Estimation of Moisture in Ores which contain no CO₂ or Organic Matter.—Weigh out 2 grms. of the sample in a small shallow dish, and place in a water oven. After being at 100° C. for two hours, it is placed in a desiccator, cooled and weighed, and the loss noted; it is again placed in water oven for half an hour and weighed. If no further loss occurs, all moisture is eliminated. $\text{Loss} \times 50 = \text{percentage hygroscopic water.}$

Estimation of Water (free and combined) in Ores containing CO₂ and Organic Matter.—Weigh out 3 grms. of the sample into a bulb tube, Fig. 8, A, and attach to a weighed CaCl₂ tube, C. This is connected with the cylinder, D, containing 36 E H₂SO₄, which serves to ascertain the rate at which the air comes through, and to absorb any moisture that

may be given off from E. E is an aspirator, and B is a tube containing CaCl_2 , which dries the air previous to its passing through A. A gentle heat is at first applied to the bulb, and air made to aspirate through gently, and the heat gradually increased to redness. After it is assured that all moisture is deposited in the CaCl_2 tube c, it is detached and carefully weighed; the increase in weight is the total water free and.

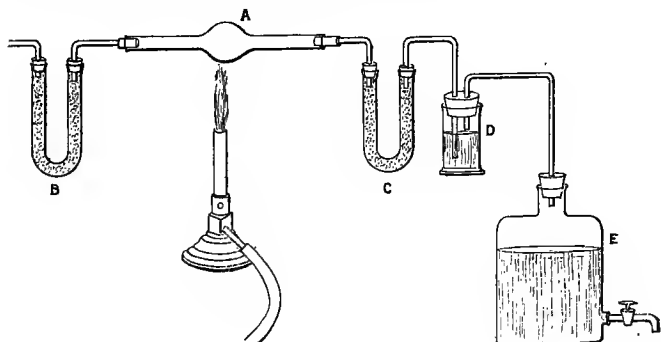


FIG. 8.

combined in 3 grms. sample. On subtracting the amount of free moisture obtained as above, the amount of combined water is obtained.

Estimation of Total Iron.—Weigh out 1 gm. of the sample into an 80 cc. beaker, add 20 cc. of 10 E HCl, and boil gently for about twenty minutes, or until no further action takes place; some ores are completely decomposable by 10 E HCl, leaving a white insoluble residue, while others are very difficult to act upon. In any case, evaporate to complete dryness and heat on a sand bath to about 130°C ., for half an hour, to render silica insoluble; cool, and boil with 10 cc. of 10 E HCl until all that is soluble is dissolved; dilute to 50 cc., filter, and wash insoluble residue with hot E HCl until free from Fe_2Cl_6 (testing washings with KCyS). Reserve filtrate, dry the

residue in water oven, ignite at a full red heat in a weighed platinum crucible, cool, and weigh. Increase in weight $\times 100$ = percentage of "Insoluble Residue." Now this insoluble residue invariably contains more or less iron, alumina, &c., and in order to estimate these add four times its weight of Fresenius' flux ($\text{Na}_2\text{CO}_3 + \text{K}_2\text{CO}_3$), fuse for twenty minutes over the blow lamp, cool, treat repeatedly with small quantities of boiling water, allow to settle, filter the clear liquid, transfer the bases to the filter with the aid of wash-bottle and feather, and wash till free from alkali. Reserve the filtrate for estimation of silica. The bases are now dissolved off the paper with 5 cc. of 8 E HCl, the paper washed till free from Fe_2Cl_6 , and the solution added to that containing the main portion of the iron. To the mixed solutions are now added a few crystals of KClO_3 (to ensure perfect peroxidation). Boil down to 50 cc. Transfer to a 250 cc. bulb flask provided with a Bunsen valve, reduce with 2 E Na_2SO_3 , and determine the iron volumetrically, as directed on page 54. This gives the total iron, *i.e.* that existing as peroxide and protoxide.

Determination of the Silica.—The solution reserved from the iron estimation for the determination of silica, is rendered neutral with 10 E HCl, 10 cc. added in excess, evaporated to dryness on the water bath, and then heated on the sand bath at about 130°C. , to render silica insoluble; 10 cc. of 5 E HCl are now added, then boiled for about ten minutes, diluted to 50 cc., allowed to settle in a warm place, the clear liquor decanted through a filter paper, the residue boiled up with 50 cc. of water, and the whole filtered. The silica, which should now be colourless, is washed till free from chlorides, with boiling water (tested with AgNO_3), dried in water oven, transferred to weighed crucible, ignited at bright red heat, cooled, and weighed. Increase in weight $\times 100$ = percentage of silica.

[The purity of the silica must be tested by evaporating with H_2SO_4 and hydrofluoric acid, and igniting. There should

be no residue left; if there is, subtract. This residue will be examined in another portion of the analysis.]

Determination of the Iron existing as Ferrous Oxide (FeO).—Many iron ores contain iron in two degrees of oxidation: the sesquioxide (Fe_2O_3) and the protoxide (FeO). The protoxide may exist in a state soluble and insoluble in HCl . To determine the FeO soluble in HCl , select a 100 cc. bulb flask, into which a rubber cork containing a tube fitted with a Bunsen valve is made to fit, weigh into it about 1 gm. of bicarbonate of soda, and then brush in 1 gm. of sample accurately weighed. Now add 12 cc. of 10 E HCl , and quickly fit in cork; the CO_2 given off by the decomposition of the bicarbonate thus drives the air out of the flask. Apply a gentle heat until the acid is in a state of incipient ebullition, which is continued until no further action is observable; dilute the solution to 200 cc., pour into a 250 cc. beaker, add 10 cc. of 10 E HCl , and titrate the solution with standard $\frac{\text{E}}{10} \text{K}_2\text{Cr}_2\text{O}_7$, in usual manner. The number of cc.'s required $\times .0056 \times 100 =$ percentage Fe existing as soluble FeO , and cc.'s required $\times .0072 \times 100 =$ percentage soluble FeO . The FeO existing in the insoluble portion is determined as follows: The insoluble residue in the above solution is filtered off through a plug of asbestos felt, free from iron, and washed with hot water; it is dried and transferred with the felt to a platinum crucible of about 25 cc. capacity. It now becomes necessary to decompose the residue so as to render the insoluble FeO soluble in HCl without undergoing peroxidation; to adopt this, the process recommended by Avery* is suitable. The crucible and its contents are placed on a platinum triangle, fixed over a hole in a special bath, Fig. 9, and 10 cc. of 10 E HCl , and 5 cc. of hydrofluoric acid (HF), poured on. It will be observed that there is a groove, G, in the top of the bath; this is partially filled with water; and the funnel, F, is made to fit into it, thus forming an

* *Chemical News*, xix., 270.

air-tight joint. A Bunsen flame is put under the bath (which is half filled with water); meanwhile a current of coal-gas is made to pass through the tube, *t*, and the heat continued until the residue and felt are completely dissolved; after which it is transferred quickly to a beaker, diluted to 200 cc., 10 cc. of 10 E HCl added, and the iron present in the ferrous state, titrated by the *standard* $\text{K}_2\text{Cr}_2\text{O}_7$, as before.

To ascertain the amount of peroxide of iron (Fe_2O_3) present

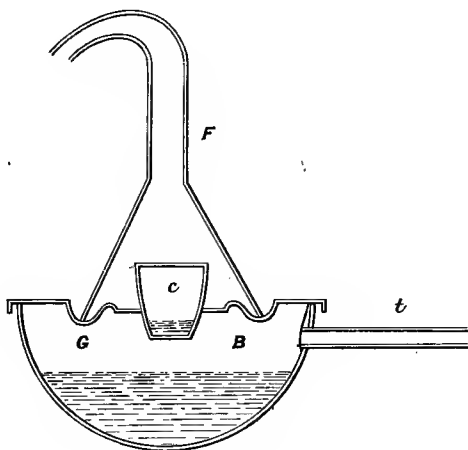


FIG. 9.

in the sample, subtract from the amount of standard $\text{K}_2\text{Cr}_2\text{O}_7$ required in the determination of total iron, the amount required for oxidation of ferrous iron in soluble and insoluble portion. This $\times .008 \times 100 =$ the percentage of Fe_2O_3 present.

Estimation of Carbonic Acid.—Some ores of iron contain a rather large percentage of combined CO_2 , such as the spathic ore, blackband ore, &c. If a preliminary test for CO_2 (made by warming some of the ore with 8 E HCl in a test-tube

and observing if there is any effervescence) gives a negative result, of course the quantitative estimation need not be proceeded with. The following is a good method for its determination when present. Fig. 10 is a sketch of the apparatus employed. Weigh out 3 grms. of the sample into the flask, D, and connect in the manner as shown below; A is a U-tube containing pieces of caustic soda, which serve to absorb CO_2 of the air that is made to pass through the apparatus by means of the aspirator, I; C is a bulb tube containing 30 cc. of 8 E HCl, which is prevented from flowing out by means of a clip

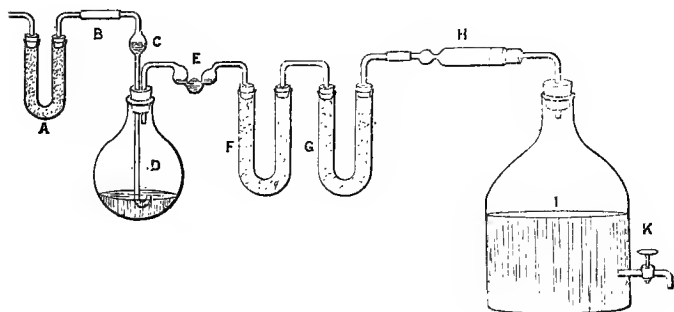


FIG. 10.

at B; E are bulbs containing a little 36 E H_2SO_4 , which serves to absorb the great bulk of moisture coming from D; F is a U-tube containing in the first limb pieces of pumice-stone, which have been soaked in a concentrated solution of copper sulphate, dried, and ignited; this serves to absorb any HCl that comes over; the second limb contains pieces of calcium chloride; G is a weighed U-tube containing in the first three quarters, small pieces of caustic potash; and the remaining quarter, pieces of CaCl_2 . This tube absorbs the CO_2 liberated, which is to be weighed before and after experiment; H is a tube containing pieces of CaCl_2 , which serve to absorb any moisture from air that may tend to come into G from I. When all is

fixed, and the tube, G, accurately weighed, open the clip and allow the acid to run on to the ore in D; after which open the tap, K, and allow a gentle stream of water to flow out; a current of air thus passes through the apparatus and sweeps the CO_2 out of D, which is absorbed in the U-tube, G. Continue the aspiration in the cold until no more effervescence is observed, and then apply a gentle heat to the flask, D; continue heating to incipient ebullition until it is assured that all CO_2 is eliminated, when the tube, G, is detached and weighed. The increase in weight, $\times \frac{100}{3}$ is the percentage of combined CO_2 present.

Determination of Alumina, Manganese Oxide, Lime, Magnesia, Potash, and Soda.—Weigh out 2 grms. of sample and heat with a mixture of 20 cc. of 10 E HCl and 6 cc. of 16 E HNO_3 , until all action has ceased, which may take about half an hour. Evaporate the solution to dryness and heat on sand-bath to about 130°C ., for about half an hour. Cool, add 10 cc. of 10 E HCl, and heat until all that is soluble is dissolved. Dilute solution to 50 cc., filter, and wash with hot $\frac{1}{10}$ E HCl until free from Fe_2Cl_6 , &c. The residue (1) is reserved. The solution is diluted to 200 cc. and 5 E Am_2CO_3 gradually added, stirring, until a slight permanent precipitate is produced. 2 cc. of 10 E HCl are now added together with 25 cc. of 5 E ammoniac acetate solution, the solution diluted to 800 cc., boiled for about ten minutes, filtered, and washed with hot water. Reserve solution, dry the precipitate in a water oven, scrape into a weighed platinum crucible, ignite, cool, and weigh; the residue may contain $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3 + \text{P}_2\text{O}_5$ (TiO_2). Deduct from this weight the amount of Fe_2O_3 and P_2O_5 present in 2 grms. of sample, as determined by the methods herein described, and the remainder $\times 50 =$ percentage of alumina in soluble portion. There may be some TiO_2 present in the residue; this should be tested for, and determined by, the method described on page 59, if any be present it is deducted, with the addition of the $\text{Fe}_2\text{O}_3 + \text{P}_2\text{O}_5$ already found, from the amount of residue, the remainder.

being the alumina. The filtrate is now poured in a $1\frac{1}{2}$ litre flask, diluted to 1 litre, 3 cc. of bromine added, stirred until all has gone into solution, 20 cc. of 20 E AmHO added, the solution heated to the boil, the hydrate of manganese filtered off, washed, and estimated as directed on page 43. Evaporate the solution to about 100 cc., add 5 cc. of 5 E AmCl, 5 cc. of 20 E AmHO, and 10 cc. of E ammonic oxalate solution, stir well, and allow to stand for about ten hours. Filter off the oxalate of lime, wash with water till free from ammoniacal salts, dry, and ignite gradually in a weighed crucible, to the highest heat of the blow-lamp. Cool and weigh the residue, which is now lime. $(\text{CaO}) \times 50 =$ percentage of lime. The solution containing the magnesia and alkalis is evaporated to dryness in a platinum dish, and the residue carefully ignited until no more fumes of ammoniacal salts are given off. Cool, add 5 cc. of water, brush in about 1 grm. of oxalic acid, evaporate to dryness as before, and ignite. The alkalis are now left as carbonates, and the magnesian chloride is converted into magnesia (MgO). Treat the residue with about 20 cc. of hot water, filter off magnesia, wash with water, dry, ignite, and weigh; this weight $\times 50 =$ percentage of magnesia (MgO).

The solution is transferred to a weighed platinum dish, and the solution made acid with 10 E HCl, evaporated to dryness without loss, and the residue carefully heated to a dull red heat for ten minutes. Transfer to desiccator to cool, and weigh the $\text{NaCl} + \text{KCl}$. Add 5 cc. of water to the residue, and then sufficient E PtCl_4 to convert the sodic and potassic chlorides into double chlorides of the alkalis and platinum. Assuming that the whole of the residue is NaCl , a calculation of the quantity required can be made. Thus every 117 parts of NaCl require 336.38 parts of PtCl_4 , forming $\text{PtCl}_4 \cdot 2\text{NaCl}$, 1 cc. of E PtCl_4 solution contains .0841 grm. of PtCl_4 . The mixture is evaporated (at a temperature of about 90°C .) over a water bath, *nearly* to dryness; after which about 15 cc. of alcohol, sp. gr. .86, are added, the dish covered with a glass plate, and allowed to stand for three hours, with occasional rotatory stirring of the contents; allow to settle before filtering, decant

the clear liquid through a weighed filter, and finally filter off the double chloride of potassium and platinum, with the aid of the filtrate and a feather. Wash with alcohol until free from the $\text{PtCl}_4 \cdot 2 \text{NaCl}$; dry at 130°C. , cool with the usual precautions, and weigh.

Calculation of the proportion of Na_2O and K_2O present :—
 $\text{PtCl}_{4,2}\text{KCl} \times .3070 = \text{KCl}$, and $\text{KCl} \times .6317 \times 50 =$ percentage of K_2O . Subtract the weight of KCl thus calculated from the weight of the $\text{NaCl} + \text{KCl}$. The remainder $\times .5302 \times 50 =$ the percentage of Na_2O present.

The reserved residue (1) is dried, brushed into a platinum crucible, 8 cc. of 10 E HCl and 4 cc. of HF added, the solution evaporated to dryness, the same quantities again added, and evaporated as before. Take up the residue in about 8 cc. of 10 E HCl , allowing to gently boil until all that is soluble is dissolved. Dilute to 30 cc., and filter if necessary. The residue, if any, must be examined for titanic acid, sulphate of barium, &c. The solution is then tested for Al_2O_3 , Fe_2O_3 (TiO_2), CaO , MnO , &c., which are estimated, if present in sufficient quantities, by the foregoing methods, taking care to keep the volume of the solutions in proportion to amounts thought to be present; and the result added to those already found in the soluble portion.

Estimation of Titanic Acid (TiO_2) in Titaniferous Iron Ores.—The following process is recommended by W. Bettel.* Weigh out 0.5 gm. of the powdered sample into a platinum crucible, mix with 6 grms. of powdered and recently-fused bisulphate of potassium, and keep in a steady fusion for about twenty minutes. Allow to cool, digest for a few hours in about 180 cc. of distilled water, and filter off any silica. Dilute the solution to about 1,200 cc., and add 4 E H_2SO_3 , until all the iron is reduced, and boil for about six hours, replacing the water as it evaporates. The precipitated titanic acid is now allowed to settle, the supernatant liquid decanted through a filter, and the residue washed

* "Crooke's Select Methods," p. 194.

by decantation and filtration with $\frac{E}{20}$ H_2SO_4 ; after which it is dried, ignited, allowed to cool, moistened with 5 E Am_2CO_3 , re-ignited, cooled, and weighed. When only very small quantities of titanitic acid are present, as in the case of some iron ores, a process similar to that described for the determination of titanium in iron is adopted.

Estimation of Phosphoric Acid (P_2O_5) and Sulphuric Acid (SO_3).—Weigh out 10 grms. of the ore into a 250 cc. porcelain dish, pour on a mixture of 45 cc. of 10 E HCl and 25 cc. of 16 E HNO_3 , boil until it is assured that all action has ceased, evaporate to dryness, heat up on sand bath, cool, re-dissolve in 50 cc. of 10 E HCl, dilute to 100 cc., filter off, wash the insoluble residue with $\frac{1}{10}$ E HCl until free from Fe_2Cl_6 , pour the filtrate into a 250 cc. graduated test mixer, and dilute to 200 cc. Divide the solution in half, pouring each 100 cc. into beakers of about 150 cc. capacity. To the one add 20 cc. of 16 E HNO_3 evaporate to the consistency of syrup on water bath, dissolve in 14 cc. of 10 E HNO_3 , dilute to 35 cc., add 40 cc. of ammoniac molybdate reagent, stir well, allow to stand at 40° C. for three hours, filter, and determine as directed on page 42. The other 100 cc. are nearly neutralized with 5 E $AmHO$, heated to boiling, 10 cc. of E $BaCl_2$ added, allowed to stand for twelve hours, and the sulphur determined as directed on page 41.

The above methods are applicable only to those iron ores which are mostly smelted in this country. For the analysis of iron ores containing arsenic, vanadium, copper, pyrites, &c., special treatises must be consulted.

The following is a table of analyses of various iron ores, used for the extraction of the metal :—*

* Thorpe, "Chemical Analysis," p. 216.

	Magnetic Ore. Dannemora.	Red Hematite. Ulverstone.	Brown Hematite. Dean Forest.	Spathic Ore. Westphalia.	Blackband Ore. Scotland.	Clay Iron Stone. Dudley.
Ferric Oxide . . .	70.23	94.23	90.05	2.75	2.72	0.40
Ferrous Oxide . . .	29.65	—	—	48.12	40.77	45.86
Manganous Oxide . . .	—	0.23	0.88	0.83	—	0.96
Alumina . . .	—	0.63	0.14	1.63	—	5.86
Lime . . .	—	0.05	0.06	1.75	0.90	1.37
Magnesia . . .	—	trace	0.20	2.29	0.72	1.85
Silica . . .	—	4.90	0.92	1.62	10.10	10.88
Carbonic Acid . . .	—	—	—	39.92	26.41	31.02
Phosphoric Acid . . .	—	trace	0.09	0.54	—	0.21
Sulphuric Acid . . .	—	0.09	traces	—	—	trace
Iron Pyrites . . .	—	0.03		0.22	—	0.10
Water . . .	—	0.56	9.22	0.45	1.00	1.08
Organic Matter . . .	—	—	—	0.39	17.38	0.90
	99.88	100.72	100.76	100.51	100.00	100.29

MANGANIFEROUS IRON ORES.

Estimation of Manganese.—The value of these ores, largely used in the manufacture of spiegeleisen, depends on the proportion of manganese present. There are difficulties in determining the manganese, as in ordinary iron ores, owing to the presence of baryta, zinc oxide, &c. The method adopted by Riley is as follows:—1 gram. of the dried ore is heated with 20 cc. of 10 E HCl until all that is soluble is dissolved. Dilute to 50 cc., filter off the insoluble residue, wash with $\frac{E}{10}$ HCl till free from soluble matter, dilute the filtrate to 300 cc., heat to boiling, add 5 cc. of 5 E H₂SO₄, stir well, allow to stand for four hours, and filter off any BaSO₄. Dilute to 1,000 cc., add 5 E Am₂CO₃ gradually, until a slight but permanent precipitate is produced; dissolve this in 3 cc. of 10 E HCl, add 20 cc. of 5 E ammoniac acetate, boil for ten minutes, filter, dissolve the unwashed precipitate,

which may contain some manganese, in 10 cc. of 10 E HCl, re-precipitate the iron as basic acetate as before, with Am_2CO_3 and $\text{AmC}_2\text{H}_3\text{O}_2$ and filter. Mix the filtrate, evaporate down to 1,200 cc., add 3 cc. of bromine, precipitate the manganese as $\text{Mn}(\text{HO})_2$ with 20 E AmHO, and estimate as directed on page 43. The ignited Mn_3O_4 should always be examined for impurities, such as baryta, zinc oxide, and lime.

Analysis of Limestones, Boiler Incrustations, and Siliceous Substances.—Weigh out 1 grm. of sample, and dry in a platinum crucible of 30 cc. capacity at 100°C . until no further loss occurs. Loss = free moisture. Add 4 grms. of white flux ($\text{K}_2\text{CO}_3 + \text{Na}_2\text{CO}_3$), fuse over blow-lamp for ten minutes, allow to cool, put the crucible and its contents into a 200 cc. beaker, pour on about 80 cc. of hot distilled water, and stir with a glass rod until the fused mass is detached from the crucible; lift the crucible out of the liquid with a pair of bone forceps, and wash it down with hot water. The solution is now boiled up, the precipitate allowed to settle, the clear liquid decanted through a filter, 50 cc. of hot water poured on the residue, boiled, and filtered as before; this operation is continued until the washings scarcely turn red litmus paper blue. The precipitate then is filtered off and washed completely with boiling water on the filter. The solution is acidulated with 10 E HCl, evaporated to dryness on a sand bath, cooled, 50 cc. of E HCl poured on to the residue and heated until all that is soluble is dissolved. It is then filtered, the insoluble residue washed till free from chlorides, the solution diluted to 100 cc., heated to boiling, E BaCl_2 added until no further precipitation takes place, allowed to stand twelve hours, and the BaSO_4 filtered off and estimated. The silica is dried, ignited in a weighed platinum crucible, cooled and weighed. It should be tested for impurities, such as TiO_2 , Al_2O_3 , &c., by evaporation with H_2SO_4 and HF, and if any residue is left it is to be examined and taken into account. The bases left on the filter, which may contain CaO, MgO, Fe_2O_3 , Al_2O_3 , are dissolved off with about

10 cc. of 10 E HCl, with addition of a few crystals of KClO_3 , the filter is washed with water, 5 cc. of 5 E AmCl added and the solution diluted to 100 cc. 20 E AmHO is now added until the solution is just alkaline, boiled, and the Fe_2O_3 and Al_2O_3 filtered off and determined by the method given on page 89. E ammonic oxalate is now added to the filtrate until no further precipitation takes place, stirred, the calcic oxalate allowed to subside, after which it is filtered off, and the CaO estimated as directed on page 90. E sodic phosphate is added to the filtrate until no further precipitation takes place (if there be no immediate precipitate about 10 cc. will be found sufficient), allowed to stand at least twelve hours, the ammonio-magnesian phosphate filtered off, the MgO determined as described on page 154, the potash and soda estimated in the filtrate as described on page 90, and the CO_2 estimated as described on page 88.

The composition of a boiler incrustation will depend upon the heat that it has been subjected to. That part which is nearest the iron and the heat will contain less carbonic acid than that on the surface, owing to its being driven off by the heat from the carbonate of lime and magnesia present, with the formation of caustic lime and magnesia, which eventually become converted into hydrates as the boiler cools down. In order to determine the combined water, about 1 grm. is ignited over a blow-lamp until it ceases to lose weight. By this means the combined water and carbonic acid and free moisture are driven off, and by subtracting the amount of carbonic acid and free moisture from the loss, as found in another portion, the proportion of combined water is obtained.

The following is an analysis of a boiler scale by the above process by Stillman :—

	Per cent.
Silica and Clay	11.70
Ferric Oxide and Alumina	2.81
Sulphate of Lime	1.69
Carbonate of Lime	5.45
Carried over	21.65

	Per cent.
Brought forward	21.65
Carbonate of Magnesia	7.36
Calcium Hydrate (CaO, H ₂ O)	13.70
Magnesium Hydrate (MgOH ₂ O)	56.37
Moisture driven of at 100° C.	0.69
Undetermined	0.20
	<hr/>
	99.97

By a slight modification many other substances can be analysed by the above process, such as clays, fire-bricks, furnace slags, &c.

ANALYSES OF SILICEOUS MATERIALS.

	Glenboig Firebrick.	Newcastle Firebrick.	Dowlais Firebrick.	Sheffield Ganister.
Silica	62.10	58.00	97.5	89.04
Alumina	33.10	36.50	1.4	5.44
Ferric Oxide	3.00	1.67	0.55	2.65
Lime	0.90	0.50	0.15	0.31
Magnesia	trace	0.90	0.10	0.17
Potash	0.90	2.12	—	—
Soda	—	.30	—	—
Loss on Ignition	—	—	—	2.30
	<hr/>	<hr/>	<hr/>	<hr/>
	100.00	99.99	99.70	99.91

ANALYSES OF VARIOUS CLAYS. (DR. MILLER).

	Washed Kaolin.			Stour- bridge Fire Clay.	Pipe Clay.	Sandy Clay.	Blue Clay.	Brick. Clay.
	Chinese.	St. Yrieix.	Cornish.					
Silica	50.5	48.37	46.32	64.10	53.66	66.68	46.38	49.44
Alumina	33.7	34.95	39.74	23.15	32.00	26.08	38.04	34.26
Ferric Oxide	1.8	1.26	0.27	1.85	1.35	1.26	1.04	7.74
Lime	—	—	0.36	—	0.40	0.84	1.20	1.48
Magnesia	0.8	trace	0.44	0.95	trace	trace	trace	5.14
Potash and Soda	1.9	2.40	12.67	—	—	—	—	—
Water	11.2	12.62		10.00	12.08	5.14	13.57	1.94
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	99.9	99.60	99.80	100.05	99.49	100.00	100.23	100.00

AVERAGE ANALYSIS OF BLAST FURNACE SLAG. (THORPE).

Silica	41.85
Alumina	14.73
Ferrous Oxide	2.63
Manganous Oxide	1.24
Lime	30.99
Magnesia	4.76
Potash	1.90
Calcium	1.15
Sulphur	0.92
Phosphoric Acid	0.15
	<hr/>
	100.32

A good mixture for lining a "Basic" Bessemer converter:—

Magnesia	37
Lime	51
Silica	8
Alumina and Oxide of Iron	4
	<hr/>
	100

ANALYSES OF MILL-FURNACE SLAGS.

	Dowlais.	Wasseral- fingen.	Sweden.	—
	Riley.	Rammels- berg.	Dugent.	Noad.
Ferric oxide	—	8.49	—	5.00
Ferrous oxide	66.01	55.36	65.83	52.50
Manganous oxide	0.19	—	0.74	—
Alumina	2.47	—	—	9.60
Lime	0.81	0.36	—	—
Magnesia	0.27	trace	trace	—
Silica	28.71	34.00	33.47	—
Sulphur	0.11	—	—	32.00
Ferrous sulphide	—	—	—	1.95
Phosphoric acid	1.22	—	—	0.25
Copper	trace	—	—	—
	<hr/>	<hr/>	<hr/>	<hr/>
	99.79	98.21	100.04	101.30
Iron, per cent.	51.34	49.0	51.20	45.34

PART III.

FUELS—SOLID, LIQUID, AND GASEOUS.

At a time when fuel has become such an expensive commodity, the consideration of its quality, heating power, and economic application, becomes a matter of great importance to engineers and steam users in general.

In determining the value of a solid fuel such as coal, coke, and patent fuel, it is necessary to estimate the amount of moisture, volatile matter, coke and its quality, sulphur, the amount of ash left on the incineration of sample, together with the calorific and evaporating power—(1) Theoretically, from a knowledge of the percentage of hydrogen, carbon, and oxygen found to be present, by an elementary organic analysis of the substance; or (2) Practically, by igniting a known weight in a calorimeter in oxygen, and ascertaining the amount of ice that has been melted, or observing the increase in temperature of a known weight of water; or (3) By ascertaining the amount of lead reduced from its oxide, by a known weight of the sample. This last method, however, is not often adopted for calorific power, the amount of lead reduced being a measure of the reducing power of a sample, rather than the amount of heat that it is capable of evolving.

The calorific and evaporating power of fuels, estimated by the foregoing methods, must only be taken relatively. The actual highest practical value of any fuel would depend very materially upon the kind of furnace used, and the regulation of an appropriate influx of air, so as to insure complete combustion. The heat carried up the flues by the products of

combustion should, if possible, be reduced to a minimum, so as to obtain the most economic results.

Liquid fuels are now coming to the front, and, judging from recent experiments made on locomotives in South Russia, where petroleum is plentiful and cheap, and coal comparatively dear, they seem to have the advantage over coal. The specific gravity, flashing point, and the calorific power obtained practically by slightly modifying the process as used in the case of coals, or by calculating it theoretically from the percentage of carbon and hydrogen found to be present, would be valuable data for guidance as to their selection. Sulphur, generally speaking, is present in so small a quantity as to be not worth consideration.

With regard to gaseous fuel, the principal heat-giving ingredients usually present are hydrogen, gaseous hydrocarbons (marsh gas, olefiant gas, &c.), and carbonic oxide. The determination of these, and the calculation of their respective calorific values on combustion, would convey an estimate of their efficiency for heating purposes.

Estimation of Moisture and Ash (Solid Fuels).—Weigh out 3 grms. of the fairly averaged powdered sample in a shallow platinum dish, dry in an air bath regulated to 105° C. for one hour, allow to cool in a good desiccator, and weigh; the loss is taken as moisture.

Coal and cokes should not be allowed to dry for more than one hour, otherwise the result obtained for moisture will be too low, owing to the oxidation of the pyrites present, by the air.

The dish with the dried sample, is now cautiously heated to redness in a muffle, until it is seen that all carbonaceous matter has been eliminated, allowed to cool in the desiccator and weighed. It is once more heated in a muffle for about five minutes, and again cooled and weighed. If the weight is unaltered from that of the last weighing, the increase in weight over the dish, is the amount of ash left by 3 grms. of sample, which can easily be expressed centesimally. The amount of

ash left by different samples is very variable, some coals showing only 7 per cent., while others as much as 20 per cent.

Estimation of Coke and Volatile Matter.—1 grm. of the sample is carefully weighed into a small porcelain crucible provided with a cover, cautiously heated to redness by a large Bunsen burner for two minutes, and then heated for an additional two minutes at a higher temperature over a gas blow-pipe; it is then allowed to cool in a desiccator and weighed. The loss = moisture + volatile matter; and the residue = coke + ash.

The coke is tested by means of a penknife, to ascertain if it is friable or compact. A little of it is placed on platinum foil and ignited, and a note made as to whether it burns freely or not.

Mr. G. E. Davis makes an interesting classification of coals, according to the amount of coke they are capable of producing:—

(1) Splint coal, burning with a long flame, and yielding from 50 to 60 per cent. of powdery or slightly caked coke.

(2) Gas coal, or coal of a bituminous nature, burning with a long flame, and yielding from 60 to 70 per cent. of fused but deeply seamed coke.

(3) Smithy coal, or true bituminous coal, burning with a long flame, and yielding 68 to 75 per cent. of fused compact coke.

(4) Caking coal, burning with a short flame, leaving from 75 to 82 per cent. of fused compact coke.

(5) Anthracite or smokeless steam coal.

Estimation of Sulphur.—Sulphur is a very deleterious constituent of coal and coke, both for boilers and metallurgical purposes. Very small quantities finding their way into “pig” iron, will render the latter unfit for steel-making, while the sulphurous vapour formed by its combustion in boilers, corrodes fire-boxes, boiler-tubes, &c.

The disagreeable odour in our underground railway tunnels,

is largely attributable to compounds of sulphur (sulphuretted hydrogen, bisulphide of carbon, &c.), mainly brought about by the distillation of fresh portions of coals, thrown on to the red-hot coke in the fire-boxes of the locomotives.

Sulphur exists in two forms in coals and cokes, being present as iron pyrites (FeS_2), technically termed "brasses," and sulphate of lime (CaSO_4).

The sulphur, present as iron pyrites, alone appears to affect the economic application of the fuel.

To determine the total sulphur present, weigh out 2 grms. of the sample and mix thoroughly with 5 grms. of pure powdered nitrate of potash. Add this mixture in small portions at a time, to 8 grms. of pure anhydrous sodic carbonate kept in a steady state of fusion in a capacious silver crucible by means of the oxidising flame of a Bunsen burner. The crucible should be tilted, and the flame kept as far from its mouth as possible, to prevent the access of sulphur compounds, from the gas. When the mixture has been fusing for about ten minutes, after the addition of the last portion of the sample, allow to cool. The crucible and its contents are now put into a 200 cc. beaker, 100 cc. of 2 E HCl poured on, digested until all that is soluble is dissolved, the crucible taken out, rinsed with water, the solution evaporated to dryness, heated on sand bath to about 130°C . to render silica insoluble, moistened with 5 cc. of 10 E HCl, diluted with distilled water to 100 cc., filtered and washed, the filtrate diluted to about 300 cc., and heated nearly to boiling. 10 cc. of an E solution of baric chloride are now added, the solution well stirred, and allowed to stand in a warm place, if possible, for about twelve hours. The supernatant liquid is now syphoned off, and the precipitated baric sulphate (BaSO_4) carefully filtered through a No. 2 Swedish filter paper, and washed till free from soluble matter with hot water. The filter paper is then spread out on a watch glass, and put to dry in a water oven: in the meantime a small porcelain crucible is carefully weighed. When the filter is dry, the BaSO_4 is brushed into it, the filter burnt separately, and its ash added to the main portion. The crucible and its

contents are then ignited at a dull red heat in the oxidising flame, allowed to cool, and weighed. Increase in weight = BaSO_4 + ash. Subtract ash, then $\frac{\text{BaSO}_4 \times .1373 \times 100}{2}$ = percentage of total sulphur.

The sulphur present as sulphate of lime is determined by boiling 5 grms. of sample with about 100 cc. of a 2 E solution of sodic carbonate; the sulphur is by this means converted into soluble sulphate of soda; dilute and filter, acidulate with 10 E HCl and estimate sulphur as above. On subtracting the sulphur thus found from the total, the amount of sulphur present as pyrites is obtained.

Estimation of Nitrogen.—Owing to the small quantity of nitrogen usually present in fuels, it is best determined volumetrically. The following is the process devised by Dumas. A combustion tube about 120 cm. long is selected, sealed at one end like a test tube, cleaned, and dried. A layer of pure bicarbonate of soda 15 cm. long is first introduced, then a layer of copper oxide 20 cm. long, then an intimate mixture of 1.5 gm. of the sample with oxide occupying 30 cm., then 30 cm. of coarse copper oxide, and 20 cm. of small copper turnings; the tube is now connected by means of a good fitting cork with the bent delivery tube, B, Fig. 11, and placed in a combustion furnace. The further end of the tube containing the bicarbonate is then gradually heated to the extent of 6 cm. Carbonic acid gas is evolved and sweeps the air out of the tube; after the gas has been coming off for a few minutes, the end of the delivery tube is dipped under mercury contained in the trough, M, and the issuing gas tested for air, by inverting a test tube filled with a 5 E solution of potassic hydrate over it. If the gas as it comes in is completely absorbed, all air has been eliminated; if not, continue the heating until the desired point is attained. The graduated tube, A, which is filled $\frac{2}{3}$ with mercury and $\frac{1}{3}$ with a 5 E solution of potassic hydrate, is then inverted over the end of the delivery tube and held in position by the clamp, K. The

combustion is now proceeded with. The fore part of the tube containing the copper is first cautiously heated to redness, and the heat gradually extended to the further end until the point where the sample ends. When no more gas comes off from the sample, the other half of the bicarbonate is heated; the second crop of CO_2 thus produced drives any nitrogen still left in the tube into A. When the volume of gas in the tube, A, is no longer diminished by the absorption of any CO_2 that

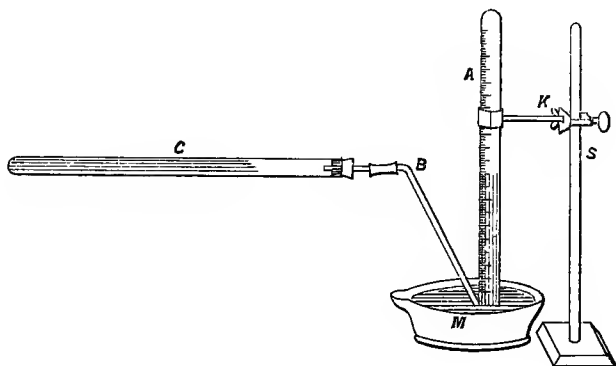


FIG. 11.

may still be present, even on shaking, the tube is transferred, by means of a small dish filled with mercury, to a deep vessel containing water. The mercury will then be displaced by water; the tube is pushed into the water until the levels of the liquids are coincident, when the volume of nitrogen is noted, together with the temperature and barometric pressure. The volume observed is reduced to 0°C . and 760 mm. pressure; and inasmuch as the gas is measured over water, allowance must be made for the reduced pressure caused by the tension of aqueous vapour at the temperature observed.

The following is an example of an actual determination of nitrogen in a sample of South Stafford coal:—

Volume of nitrogen observed	25 cc.
Temperature	15° C.
Barometric pressure	758 mm.

The tension of aqueous vapour at 15° C. is equivalent to 12.677 mm. of mercury.

Taking 1 cc. of nitrogen at 0° C. and 760 mm. pressure as weighing .0012544 grm., then the percentage of nitrogen by weight in the sample is—

$$\frac{25 \times 273 \times (758 - 12.677) \times .0012544 \times 100}{(273 + 15) \times 760 \times 1.5} =$$

1.943 per cent. nitrogen.

Estimation of Carbon and Hydrogen.—The principle of the method adopted for the determination of carbon and hydrogen depends upon the fact that when a fuel is burnt in excess of air or oxygen, or any oxidizing substance, the carbon is oxidized into carbonic acid gas (CO_2), while the hydrogen is converted into water (OH_2), evolved as steam. A known weight of the sample being taken, it is ignited with chromate of lead, or with copper oxide and oxygen or air; the products of combustion, CO_2 and OH_2 , are absorbed by appropriate reagents, and weighed separately, from which the amounts of carbon and hydrogen present are deduced by a simple calculation. The following is the *modus operandi* of the process :—

Select a wrought-iron tube 20—22 mm. in diameter and 115 cm. long; oxidize inner surface of tube by heating it to redness in a combustion furnace, and passing a current of steam through. A layer of recently ignited coarse oxide of copper, about 20 cm. long, is pushed into the middle of the prepared tube and kept in position by plugs of copper gauze, placed one each side; a sheet-iron boat about 30 cm. long, is nearly filled with recently fused and powdered chromate of lead, and introduced into the tube, which is placed in a combustion furnace and heated below the fusing point of the chromate, a current of dry air being passed through, to rid the tube of any moisture. The gas is then put out and the tube plugged and cooled. The boat is then taken

out, and from .3 to .5 grm. of the powdered sample, free from water, is quickly and thoroughly mixed with the chromate, and replaced in the tube; a similar boat filled with recently reduced metallic copper is introduced at the other end of the tube, and the whole placed into the furnace and coupled up to the necessary desiccating and absorption apparatus. Fig. 12 shows the apparatus ready for a combustion.

b is a cylinder containing a 5 E solution of potassic hydrate, which absorbs the great bulk of CO_2 present in the oxygen or air used for the combustion; it is coupled to a gas holder by

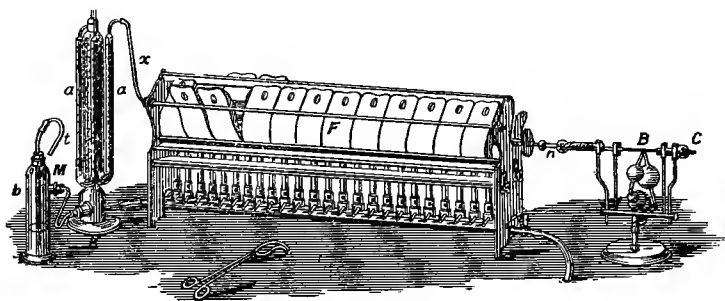


FIG. 12.

means of the tube *t*, and to the cylinder, *k*, which is filled with fragments of soda lime, which completes the absorption of CO_2 . The U tubes *a a*, are filled with dry granulated calcium chloride, which completely absorbs atmospheric moisture. They are connected to the combustion tube by means of the pipe, *x*; the bulb tube, *n*, contains dry calcic chloride which serves to absorb the water brought about by the combustion of the hydrogen in the sample. The bulbs, *B*, contain a 5 E solution of potassic hydrate, which absorbs the CO_2 produced by the combustion of the carbon. *c* is a small tube containing calcium chloride, which serves to retain any moisture carried mechanically from *B* by the issuing gas.

Before proceeding with the combustion, *n* and *B* are accurately weighed separately and attached to tube as in sketch;

all joints having been insured air tight, the gas is turned on at the end of the tube containing the reduced copper, and a gentle current of air made to pass through the apparatus, when it is at a dull red heat. The gas is then gradually turned on until the boat containing the sample is reached. Care is here required, and the heat should be gradually raised to full redness, when the chromate will fuse, and the sample will soon be completely oxidized. After it has been insured that the combustion is complete, the calcium chloride tube, *n*, and potash bulbs, *B*, are detached and weighed. The increase in weight of the CaCl_2 tube, multiplied by $\cdot 1111$, gives the amount of hydrogen in quantity of sample taken, and the increase in weight of the bulbs, *B*, multiplied by $\cdot 27273$ gives the amount of carbon present in the quantity of sample taken.

In estimating the carbon and hydrogen in non-volatile liquid fuels, the two boats are filled with copper oxide, the weighed liquid absorbed in one of the boats, and the combustion proceeded with as above; or if it be a volatile hydrocarbon it is weighed into a small thin glass tube, with a loose stopper, and dropped into the boat, due care being taken that the combustion is not hurried, otherwise vapour will escape unoxidized.

The following is an example of the results obtained for carbon and hydrogen in a Scotch bituminous coal, when $\cdot 5$ grm. was taken :—

Weight of CaCl_2 tube before combustion	.	.	.	30.4562
„ „ after „	.	.	.	30.6884
Water absorbed				<u>.2322</u>
$\cdot 2322 \times \cdot 1111 \times 2 \times 100 = 5.159$ per cent. hydrogen.				
Weight of potash bulbs before combustion	.	.	.	52.0318
„ „ after „	.	.	.	53.4296
CO_2				<u>1.3978</u>
$1.3978 \times \cdot 27273 \times 2 \times 100 = 76.244$ per cent. carbon.				

Estimation of Oxygen.—There is no ready method for the direct determination of oxygen in complex organic compounds.

It is as a rule estimated by difference. Having a knowledge of the percentage of all other ingredients present in the sample, adding up and subtracting from 100, the remainder is taken as oxygen.

The Specific Gravity of Coals, &c.—It is often desirable to know the amount of space that a given weight of coal will occupy, and the determination of the specific gravity thus becomes necessary.

To obtain this a small flask provided with a thermometer stopper, and holding a definite weight of water at a known temperature—usually 15° C., which is previously accurately ascertained—is taken, and 2 to 3 grms. of the sample weighed into it; water is then added, and the coal allowed to soak in it for some time, so as to eliminate air from the pores. The bottle is then filled with water at the standard temperature and again weighed.

The specific gravity is obtained as follows:—

$$\begin{aligned}\text{Let } W &= \text{weight of sample in air} \\ R &= \text{weight of flask + water} \\ R_1 &= \text{weight of flask + water + sample}\end{aligned}$$

Then,

$$\text{Specific gravity} = \frac{W}{W + R - R_1}$$

The weight of a cubic foot of the sample in pounds is obtained by—

$$\log. \text{ specific gravity} + 1.79588 = \log. \text{ weight of cubic foot.}$$

The number of cubic feet in a ton

$$= 1.55437 - \log. \text{ specific gravity} = \log. \text{ cubic feet.}$$

It is very important in determining the specific gravity of coals and cokes, to insure that all air has been driven out of the sample by the water before diluting and weighing. An example may be given of the error that would be involved if this precaution were not taken. Mr. Crookes, F.R.S., obtained

the following: 2.76 grms. of coal gave a specific gravity of 1.309 at 64° F., immediately after filling the flask with water; after soaking twelve hours the specific gravity had increased to 1.328 for the same temperature. So that the latter determination would make a cubic foot of this coal weigh 82.76 lbs., and the former only 81.58, or 1.18 lbs. less.

Specific Gravity of Liquid Fuel.—The specific gravity of liquid fuels can in the majority of cases be determined at 15° C. by the hydrometer in the usual manner. It sometimes happens, however, that liquid fuels are too thick to obtain an accurate result by this means, and it has to be determined by the specific gravity bottle; or if too thick for this, by placing a drop in a cylinder of alcohol at 15° C., and adding water until it remains stationary in any part of the fluid in which it is placed, a glass rod being used for the purpose. The specific gravity of the fluid is then taken with the hydrometer, and the specific gravity of the sample is thus attained. (See page 189).

The Flashing Point.—The temperature at which the vapour coming off from liquid fuels ignites when mingled with air, on the application of a flame, is of some importance. The lower the temperature at which the vapour is capable of igniting, the more care, of course, will be required with its handling, storage, transport, &c.

The flashing point can be determined in the manner directed on page 206. But in cases where the flash of the oils is above the temperature of boiling water, the oil cup is to be placed in an air bath and heat applied direct with a flame, which is kept under the bath the whole of the time, the rest of the process being conducted as described.

Determination of the Calorific Value of Solid and Liquid Fuels by Thompson's Calorimeter.—In this method, which is now most extensively used in estimating the heating power of fuels, a known weight of sample is ignited with an oxygen mixture in a copper cylinder, in a known

weight of water, the temperature of which is first accurately observed. From the increase of temperature of the water, due to the combustion of sample, the comparative heating and evaporating power can be deduced.

The French unit of heat, is the amount of heat necessary to raise the temperature of 1 grm. of water through 1° C., or more correctly from 0° to 1° C. The calories as here expressed are the number of grms., lbs., or any unit weights of water raised 1° C. by the combustion of 1 grm. or 1 lb. &c., of the sample. These can be converted into British Thermal Units :

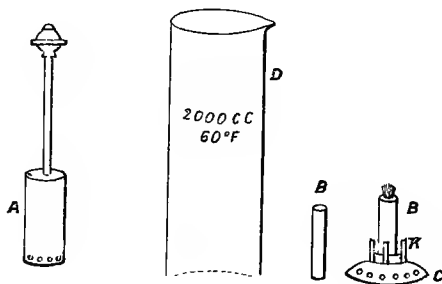


FIG. 13.

viz., lbs. of water raised 1° F. by 1 lb. of sample, by multiplying by $\frac{9}{5}$.

For coals and cokes, 2 grms. of the finely powdered and dried sample, are thoroughly mixed with 26 grms. of a finely powdered and dry mixture, of chlorate of potash, 3 parts, and nitrate of potash, 1 part, on a sheet of glazed paper. By means of a flexible steel spatula, the mixture is introduced in small quantities at a time into the copper cylinder B, Fig. 13, each addition being pressed with the same pressure, with the rounded end of a test-tube, so that a fairly uniform combustion can be relied upon. When all has been brushed in, a short length of fuse (prepared by soaking lamp cotton in a strong solution of nitre, and drying) is pushed into the mixture, and

about half an inch allowed to protrude. It is then fixed into the brass stand C. The glass cylinder D, having been charged with 2,000 cc. of water, the condenser A is fixed over B into C, held firmly by the clutches K. The whole is then placed into D, and moved up and down in the water until the temperature of the latter is fixed. The temperature is recorded by a very delicate thermometer graduated to $\frac{1}{100}$ of a degree Centigrade.

The temperature of the room is usually higher than the water, so that a little hot water or ice (as the case may demand) must be added, until the differences are about as follow :—

Room at ° C.	27	23	20	16	13	10	6
Water should be	21	18	15	12	10	8	5
Differences	6	5	5	4	3	2	1

When this is accomplished the apparatus is lifted out of the cylinder D and the condenser detached. A light is then applied to the fuse, the condenser *quickly* replaced, and the whole plunged into the water; when gas appears through the holes at the bottom of A the time is noted, and an observation made as to the regularity of the combustion. When the combustion is at an end, which should in most cases occupy not less than 60 seconds, the stopcock of A is opened and a wire forced down the pipe to clear it; the whole is then moved up and down in the liquid with the thermometer until the temperature has attained its maximum, and the increase is then noted.

Often there is a small quantity of sample that has escaped combustion; and to make allowance for this, the liquid is made acid with hydrochloric acid, and evaporated to a small bulk in a porcelain basin. The residue is filtered off, washed, dried, brushed into a tared crucible, dried and weighed; it is then ignited in a muffle, cooled, and again weighed; the loss is assumed to be the carbon and hydrogen unburnt.

To ascertain the temperature this would raise the water if completely burnt in the calorimeter: Let V = combustible matter minus water in 2 grms. sample, C amount of carbon and hydrogen found to be unburnt, and T_1 the rise in tempera-

ture in calorimeter, then the temperature T_2 corresponding to C will not be far short of,

$$\frac{T_1 \times C}{V}$$

The following is an example of the determination of the heating power of a sample of Welsh steam coal by the above process :—

Temperature of room	17.25° C.
„ „ water before combustion	13.20° C.
„ „ „ after „	20.30° C.
Increase in temperature $20.3 - 13.2$	$= 7.10^\circ \text{ C.}$
Allowance for temperature of C and H unburnt	$.21^\circ \text{ C.}$
	<hr/>
	7.31° C.
Absorption of heat by calorimeter $\frac{1}{10}$	$= .73^\circ \text{ C.}$
	<hr/>
Total	8.04° C.

$8.04 \times 1,000 = 8,040$ calories — that is, pounds of water heated 1° C. by 1 pound of sample.

The latent heat of steam being 537 thermal units, the evaporative power (lbs. of water evaporated at 100° C. (212° F.) by 1 lb. of coal) becomes $\frac{8040}{537} = 14.97$.

On determining the heating power of substances rich in hydrogen—such as patent and liquid fuels—the oxygen mixture is diluted with from .5 to 3 grms. of dry kaolin clay. The combustion is sometimes difficult to start; in most cases, however, this may be overcome by the employment of a little gunpowder or coal mixture with the fuse, the calorific value of which has been previously ascertained; and the temperature corresponding to the amount taken, must be subtracted from the increase of temperature observed.

Calculation of the Theoretical Calorific Values of Solid and Liquid Fuels from the Chemical Analysis.—Carbon and hydrogen are the only elements in solid and liquid

fuels that may be considered the source of their heating efficiency, consequently the amount of heat that would be expected from them would be thought to depend entirely upon the respective amounts of these elements present in the fuel. The quantity of oxygen present, however, has to be taken into account, which is assumed to be combined with its equivalent of carbon or hydrogen as the case may be, and renders so much of the latter incapable of generating heat.

The amount of heat rendered ineffective by the quantity of oxygen present, will depend upon whether the latter is supposed to be combined with carbon, or hydrogen. The amount of carbon rendered latent by a given quantity of oxygen, would be three times the amount of hydrogen rendered latent by the same quantity of oxygen.

The heat given out in the combustion of hydrogen is always the same, but in the case of carbon it depends upon whether it is oxidized to its maximum as carbonic acid (CO_2) or to its minimum carbonic oxide (CO), so that carbon can have two calorific values according whether the product of combustion be CO or CO_2 .

In practical working, great loss of heat would be entailed if the carbon were not oxidized to its full, as the following shows :—

	Calories.
The heat generated by the combustion of 1 grm. carbon to carbonic acid is	= 8080
While the heat generated by the combustion of the same weight of carbon to carbonic oxide is	= 2473
Loss of heat by production of CO	= 5607

From accurate determinations made by Favre and Silbermann it has been found that the amount of heat generated by the combustion of hydrogen is 4.265 times as great as the heat given out by the combustion of the same weight of carbon to carbonic acid.

The relative calorific power of fuels may be deduced from the following formulæ :—

- (1.) Fuel containing carbon only . . . $p = C$.
 (2.) Fuel containing carbon and hydrogen $p = C + 4.265 H$.
 (3.) Fuel containing carbon, hydrogen, and oxygen . . . $p = C + 4.265 (H - \frac{1}{8} O)$

where, p = relative calorific power

C , H and O = amounts of carbon, hydrogen, and oxygen present in 1 part of fuel.

If it be required to express the calorific power of a fuel in heat units then—

- (1.) $p = 8080 C$.
 (2.) $p = 8080 C + 34462 H$.
 (3.) $p = 8080 C + 34462 (H - \frac{1}{8} O)$.

The following table gives the calorific values of several substances as calculated from the above formulæ :—

Fuel.	Composition of fuel.				Relative calorific power.		Weight of Water heated from 0° to 100° C.	Wgt. of Water at 100° C. converted into Steam.
	Carbon.	Hydrogen.	Oxygen.	Ash.				
Hydrogen . . .	—	1.00	—	—	4.265	34,462	344.62	62.658
Marsh gas . . .	0.75	0.25	—	—	1.816	14,675	146.75	26.682
Olefiant gas . . .	0.875	0.143	—	—	1.466	11,849	118.49	21.543
Welsh coal . . .	0.838	0.048	0.041	0.049	1.020	8,241	82.41	14.983
Newcastle coal . . .	0.821	0.053	0.057	0.038	1.017	8,220	82.20	14.945
Carbon . . .	1.000	—	—	—	1.000	8,080	80.80	14.691
Scotch coal . . .	0.785	0.056	0.097	0.040	0.973	7,861	78.61	14.292
Derbyshire coal . . .	0.797	0.049	0.101	0.026	0.956	7,733	77.33	14.060
Lancashire coal . . .	0.779	0.053	0.095	0.049	0.955	7,717	77.17	14.031
Kiln-dried peat . . .	0.600	0.060	0.307	0.020	0.694	5,640	56.40	10.254
Air-dried peat . . .	0.461	0.046	0.246	0.015	0.526	4,250	42.50	7.727

In determining the thermal effect of fuels, from their percentage composition, when made to burn in air, corrections have to be made for the latent heat of water, produced by the combustion of the available hydrogen ; and the specific heats of the carbonic acid, water vapour, nitrogen, and air, have also to be

taken into account. The following is a formula for arriving at the thermal effect of a fuel when completely oxidized in air :—

$$T = \frac{cC + c'H - lW}{S \cdot 3 \cdot 67 C + 9 H + S' W + S'' N + S''' A}$$

Here T = increase of temperature produced by combustion.

C and H = quantities of carbon and hydrogen available in 1 part fuel.

W = water produced by 1 part fuel.

l = latent heat of water.

$S S' S'' S'''$ = specific heats of carbonic acid, water vapour, nitrogen and air.

N = nitrogen in quantity of air necessary for complete combustion of fuel.

A = any additional amount of air supplied for combustion.

The result obtained by the above formula expresses the highest heat attainable as compared with carbon burnt to its highest oxide under the best conditions. The amount of heat generated practically, however, is usually less than what should be obtained by calculation as above; this is due to a variety of causes, such as imperfect combustion, loss of fuel as smoke, imperfectly oxidized cinders, &c.

Rankine adopts as his unit, the weight of fuel required to evaporate 1 lb. of water at 212° F. under a pressure of 14.7 lbs. per square inch; this being equivalent to 966 British Thermal Units or 537 Centigrade Units. The results are obtained as follows :—

Let E be the corrected and reduced evaporation,

e the weight of water evaporated,

T_1 the standard boiling point, 212° F. ($= 100^{\circ}$ C),

T_f the temperature of feed water,

T_b the actual boiling point observed;

then,

$$E = e \left\{ 1 + \frac{T_1 - T_f + 0.3(T_b - T_1)}{966 \text{ F. or } 537 \text{ C.}} \right\}$$

The result is the number of times its own weight of water which a fuel would evaporate at the standard temperature, if no loss of heat occurs; but as there is always some loss of heat, the efficiency of the furnace is expressed by the ratio, $\frac{E \text{ (Available)}}{E \text{ Total}}$, which, if no waste occurred, would be $= 1$.

as the sum of that of the hydrogen and carbon present, assuming that when oxidized with the theoretical proportion of air, each lb. of carbon evaporates 11.359 lbs. of water at 155° C., and each lb. of hydrogen 41.895 lbs. of water at 155° C., into steam at 100° C. The results obtained by this method are given in the following table. Column 5 gives the evaporation duty when the furnace gases are discharged at 600° F. above the temperature of the air supplied to the furnace.

	Carbon.	Hydrogen.	Oxygen.	Evaporative Power, lbs. water at 100° C.	Evaporative Duty, lbs. water at 155° C.
Phenol	76.6	6.40	17.00	12.2437	10.5025
Cresol	77.77	7.41	14.82	13.0096	11.1632
Naphthalin . .	93.75	6.25	—	15.4350	13.0751
Anthracine . .	94.38	5.62	—	15.2417	13.2675
Xylol	90.56	9.44	—	16.5866	14.2415
Cumol	90.00	10.00	—	16.7838	14.4126
Cymol	89.55	10.45	—	16.9422	14.5500

It is computed, generally speaking, that in average practical working, 1 lb. of liquid fuel would not be likely to evaporate more than 16 lbs. of water as steam.

An example of Paul's method of obtaining the *effective* heat is as follows:—

COMBUSTION OF 1 LB. OF CARBON.

	Heat Units.	Equivalent Evaporation of Water.	
		At 212° F.	At 60° F.
Total heat of combustion	14,500	15	
Available heat	14,500		
Waste by furnace gases at 600° F. .	3,480	3.6	
Effective heat	11,020	11.4	9.8

COMBUSTION OF 1 LB. OF HYDROGEN.

	Heat Units.	Equivalent Evaporation of Water.	
		At 212° F.	At 60° F.
Total heat of combustion	62,032	64.2	
Latent heat of water vapour	8,695		
Available heat	53,337	11.9	
Waste heat of furnace gases	11,520		
Effective heat	41,817	42.3	38

The following are the results obtained per lb. of two kinds of liquid fuel, A and B:—

A containing 86 per cent. carbon and 14 per cent. hydrogen.

B " 75 " " 25 " "

A.

Carbon.	Hydrogen.	Total Heat of Combustion.	Equivalent Evaporation of Water.	
			At 212° F.	At 60° F.
86	14	$\times 14500 = 12470$ $\times 62032 = 6684$ <hr/> 21154	21.9	18.8
Furnace Gases.		Heat units in Furnace Gases.		
	lbs.		2.2	
Carbonic acid	3.16	411		
Water vapour	1.26	359	4.8	
Nitrogen	11.45	1683		
Surplus air	14.37	2124	15.8	13.6
	30.74	4577		
Total heat of combustion		21154	1.3	
Latent heat of water vapour		1217		
Available heat		19937	4.8	
Waste in furnace gases		4577		
Effective heat		15360	21.9	
Theoretical evaporating power				

B.

Carbon.	Hydrogen.	Total Heat of Combustion.	Equivalent Evaporation of Water.	
			At 212° F.	At 60° F.
·75	·25	$\times 14500 = 10775$ $\times 62032 = 15508$ <hr/> 26283	27·1	23·1
Furnace Gases.		Heat units in Furnace Gases.		
Carbonic acid . . .	lbs. 2·75	358		
Water vapour . . .	2·25	641		
Nitrogen . . .	13·39	1968		
Surplus air . . .	17·39	2483	2·6	
	35·78	5450		
Total heat of combustion . . .		26283		
Latent heat of water vapour . . .		2174	2·2	
Available heat		24109		
Waste in furnace gases		5450	5·6	
Effective heat		18659	19·3	16·6
Theoretical evaporating power			27·1	

GASEOUS FUEL.

The great progress that has recently been made in the manufacture and application of gas, suitable for fuel, metallurgical and domestic heating purposes, &c., necessitates a ready method by which its analysis and heating power can be quickly deduced with fair practical accuracy.

For the very accurate and scientific analysis of complex gas mixtures, delicate processes, such as Frankland and Ward's, would have to be resorted to, which would, generally speaking, be far too tedious and elaborate for practical working purposes. In metallurgical works, water gas works, &c., it is often neces-

sary to have several complete technical analyses of gas in a single day, in order to give an idea of the economic working of processes, &c.

The apparatus which seems to commend itself for quick working and reasonable accuracy, is that devised by Elliot, of

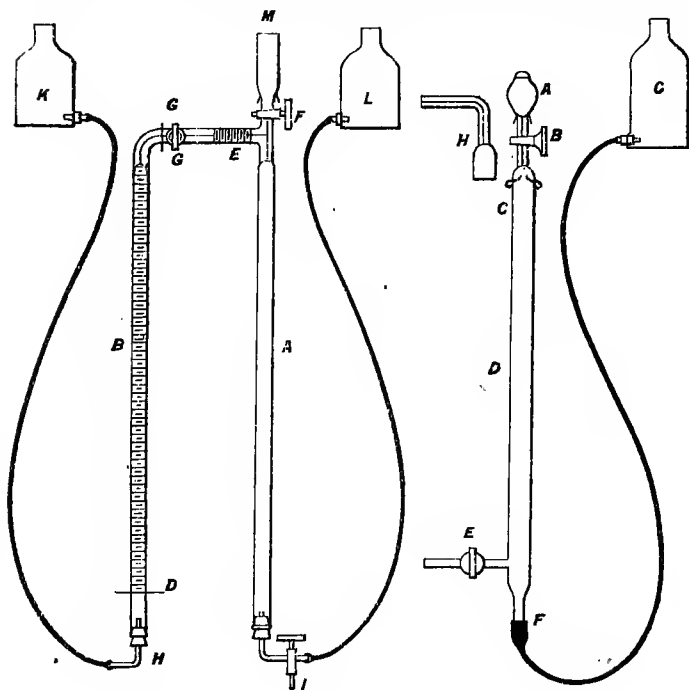


FIG. 14.

FIG. 15.

which a sketch is here given. B, Fig. 14, is a tube graduated to 100 cc. in $\frac{1}{10}$ th cc. The stop-cock I is a three-way tube with a delivery tube through its stem. The bottles K and L hold about a pint each. M is a portable funnel ground to fit above F and holds 60 cc. E is a rubber tube joining A and B.

Before starting an analysis, the tubes A and B are filled with

water from the bottles K and L, by the manipulation of the stop-cocks C, F, and I; when water rises into the funnel M, and all air eliminated, F and G are turned off, the funnel M removed, and the tube containing the gas for analysis attached in its place; the bottle L is now slowly lowered until A is filled with gas, and the stop-cock F is then closed; remove the gas tube from F, replace the funnel M, raise the bottle L, lower the bottle K, and open the stop-cock G. The gas is thus driven into the graduated tube B. Lower the bottle K so that the level of the water therein is in a line with the zero mark D. The gas is then adjusted to the zero mark D by the bottle L, the stop-cock G closed, and the temperature and pressure recorded.

After the surplus gas has been displaced from the tube A by raising L and opening F, by manipulating the bottles and stop-cocks the gas is drawn into A. Close the cocks, lower the bottle L, and fill the funnel M with a solution of 5 E potassic hydrate. Cautiously open F and allow the liquid to flow into the tube A, always leaving, however, about 10 cc. in M. On allowing to stand until no further diminution in volume occurs, due to the absorption of carbonic acid (CO_2) present in the gas, the residual gas is transferred to the tube B and measured, the temperature and pressure being noted; the loss on the original volume is CO_2 . Empty the tube A, wash out with water, and refill with water as before. The gas from B is transferred to it, the funnel M is half filled with water, and a few drops of bromine added; this is run into the tube until fumes of bromine are seen to be mixed with the gas.

On allowing to stand until no further contraction occurs, due to the absorption of ethylene and other illuminants, some of the potash used for the absorption of the CO_2 is added; this absorbs the excess of bromine. When this is complete the gas is measured as before, the loss in volume being put down to illuminants.

After A has been cleared out and refilled with water, the gas is again brought into it as before; the funnel M is filled with a solution of 5 E potassic hydrate, to which has been added about 3 per cent. of pyrogallic acid; this is run into the tube

and the gas allowed to remain over it, until any oxygen present is completely absorbed; it is then measured as before. Loss = oxygen. Clean out A, withdraw the gas from B into it, fill M with 10 E hydrochloric acid, containing 25 per cent. of cuprous chloride, and allow to stand till no further diminution in volume occurs, and measure as before; the loss is due to carbonic oxide (CO).

The residual gas may now contain marsh gas, hydrogen, and nitrogen; and to determine the proportion of these it will be necessary to explode them with oxygen by an electric spark. This is accomplished by means of the explosion tube, Fig. 15, graduated to 100 cc. in $\frac{1}{10}$ cc. to within 2 inches from E, the stop-cock B being the zero point. The funnel A is portable like M in Fig. 14; at C there are two platinum wires fused in, which are connected with an induction coil. The bent tube H is made to fit over the stop-cock B when the funnel A is removed, which serves to facilitate the transfer of gas. Before starting, remove the funnel M in the absorption tube, and fix in its place a bent tube like H. The gas having been transferred to A, the explosion tube is placed near it, and the bent tube H is attached to a piece of rubber tube, long enough to reach to the corresponding bent tube of the explosion tube.

The explosion tube is now filled with water from the bottle G, to the end of the tube H over the stop-cock B. Fill the piece of tubing with water, and connect the two bent tubes H with it. Now turn the three-way cock I, so that the bottom of the tube A is closed. Open B and F, and by the aid of the bottles draw in 20 cc. of the gas into the explosion tube, manipulating with the bottle G, and close the stop-cocks. Connect the inlet tube E with a gas-holder of oxygen under pressure, and introduce about 20 cc. into the tube, and mix; level off and note total volume. Place the bottle G below F to expand the gases, pass the spark, and with a click the explosion is complete. Allow the flush of heat to debate, and observe the contraction after levelling with G.

By removing the bent tube H and fixing on the funnel A, the amount of CO₂ produced can be ascertained by introducing

the solution of potassic hydrate, and observing the diminution in volume. The formula for the calculation of the proportion of hydrogen, marsh gas, and nitrogen present in the 20 cc. of residual gas taken (which must afterwards be calculated on the original gas taken), becomes :—

Let C = contraction ;
 D = carbonic acid formed ;
 X, Y, Z = H, CH₄, and N respectively ;

then—

$$\begin{aligned} X &= \frac{2C - 4D}{3} \\ Y &= D \\ Z &= \frac{3A - 2C + D}{3} \end{aligned}$$

In working with the apparatus, the analysis should be performed in a room where the temperature would remain uniform during the analysis, and care should be taken that the water, chemicals, &c., are at the same temperature as the room.

Inasmuch as a complete analysis can be made by this process in the course of an hour, the temperature and pressure, if suitable precautions be taken, would rarely be altered during the readings ; and the original gas being usually saturated with moisture, no correction would be necessary for the tension of aqueous vapour, since it is measured over water.

Calorific Value.—The heating power of a gaseous fuel containing carbonic acid, hydrogen, marsh gas, olefiant gas, nitrogen, carbonic acid, and aqueous vapour, Bunsen deduces as follows :—

$$A = 3000 [x \cdot k \cdot 0.57 + 1.5 \cdot h \cdot 8 + 1.1 \cdot g \cdot 4 + 1.17 \cdot o \cdot 3.43] \\ - 550 [g \cdot h + 2.25 \cdot g + 1.29 \cdot o + w]$$

Where K = amount of CO

H =	„	H
g =	„	CH ₄
o =	„	C ₂ H ₄
n =	„	N
w =	„	Aqueous vapour
k =	„	CO ₂

For the pyrometric heating effects of gaseous fuel burnt in air the following formula is used :—

$$T = \frac{A}{Q(K)S + Q(w)S' + Q(n)S''}$$

$$Q(K) = K + 1.57 \cdot h + 2.75 \cdot g + 3.14 \cdot o$$

$$Q(w) = w + 9 \cdot h + 2.25 \cdot g + 1.29 \cdot o$$

$$Q(n) = n + 3.33 (0.57 \cdot h + 8 \cdot h + 4 \cdot g + 3.43 \cdot o)$$

$SS'S''$ = specific heats of CO_2 , OH_2 , and N respectively. A much higher temperature would be produced if burnt in pure oxygen, as the following calculation by Bunsen by the above formula shows :—

	In oxygen.	In air.
Carbon	9873° C.	2458° C.
Carbonic oxide	7067	3042
Olefiant gas	9187	5413
Marsh gas	7857	5329
Hydrogen	8061	3259

METHOD OF ASCERTAINING THE THERMIC VALUE OF A GAS COMPARED WITH COAL.

(*Ford, Jour. I. and S. Inst.*)

A natural gas from the Pittsburg district has the following average chemical composition :—

Carbonic acid	0.60 per cent.
Carbonic oxide	0.60 „
Oxygen	0.80 „
Olefiant gas	1.00 „
Ethylic hydride	5.00 „
Marsh gas	67.00 „
Hydrogen	22.00 „
Nitrogen	3.00 „

Now by the specific gravity of these gases we find that 100 litres of this gas will weigh 64.8585 grms., thus :—

Marsh gas	.	67.0 litres weighs	48.0256 grms.	
Olefiant gas	.	1.0 "	"	1.2534 "
Ethylic hydride	.	5.0 "	"	6.7200 "
Hydrogen	.	22.0 "	"	1.9712 "
Nitrogen	.	3.0 "	"	3.7632 "
Carbonic acid	.	.6 "	"	1.2257 "
Carbonic oxide	.	.6 "	"	0.7526 "
Oxygen	.	.8 "	"	1.1468 "
Total	.	.	.	64.8585 "

Then if we take the heat units of these gases, we will find

Marsh gas	.	48.0256 grms. contain	627,358 heat units.	
Olefiant gas	.	1.2534 "	14,910 "	
Ethylic hydride	.	6.7200 "	77,679 "	
Hydrogen	.	1.9712 "	67,921 "	
Carbonic oxide	.	.7526 "	1,808 "	
Nitrogen	.	3.7630 "	— "	
Carbonic acid	.	1.2257 "	— "	
Oxygen	.	1.1468 "	— "	
		<u>64.8585</u> "	<u>789,694</u> "	

64.858 grms. are almost exactly 1,000 grains, and 1 cubic ft. of this gas will weigh 267.9 grains; then the 100 litres or 64.8585 grms. or 1,000 grains are 3.761 cubic ft.

3.761 cubic ft. of this gas contain 709,694 heat units, and 1,000 cubic ft. will contain 210,069,604 heat units. Now 1,000 cubic ft. of this gas will weigh 265,887 grains, or 38 lbs. avoirdupois.

We find that 64.8585 grms. or 1,000 grains of carbon contain 524,046 heat units, and 265,887 grains or 38 lbs. of carbon contain 139,398,896 heat units. Then 57.25 lbs. of carbon contain the same number of heat units as 1,000 cubic ft. of the natural gas, viz., 210,069,604.

Now, if we say that coke contains in round numbers 90 per cent. of carbon, then we will have 62.97 lbs. of coke equal in heat units to 1,000 cubic ft. of natural gas.

Then, if a ton of coke, or 2,000 lbs., cost 10s., 62.97 lbs.

will cost 4d., or 1,000 cubic ft. of gas is worth 4d. for its heating power.

We will now compare the heating power of this gas with bituminous coal, taking as a basis a coal slightly above the general average of the Pittsburg coal, viz.—

Carbon	.	.	.	82.75	per cent.
Hydrogen	.	.	.	5.31	"
Nitrogen	.	.	.	1.04	"
Oxygen	.	.	.	4.64	"
Ash	.	.	.	5.31	"
Sulphur	.	.	.	0.95	"

We find that 38 lbs. of this coal contain 146,903,820 heat units. Then 54.5 contain 212,069,640 heat units, or 54.4 lbs. of coal are equal in their heating power to 1,000 cubic ft. of natural gas. If our coal costs us 5s. per ton of 2,000 lbs., then 54.4 lbs. will cost 1.632 pence, and 1,000 cubic ft. of gas will be worth for its heat units 1.632 pence.

As the price of coal increases or decreases, the value of the gas will naturally vary in like proportions. Thus with the price of coal at 10s. per ton, the gas will be worth 3.264 pence per 1,000 cubic ft.

If 54.4 lbs. of coal be equal to 1,000 cubic ft. of gas, then 1 ton, or 2,000 lbs., will be equal to 36,764 cubic ft. or 2,240 lbs. of coal will be equal to 40,768 cubic ft. of natural gas.

If we compare this gas with anthracite coal, we find that 1,000 cubic ft. of gas are equal to 58.4 lbs. of this coal, and 2,000 lbs. of coal are equal to 34,246 cubic ft. of natural gas. Then if this coal costs 26s. per ton, 1,000 cubic ft. of natural gas will be worth 9½d. for its heating power.

Tables of Practical Results and Analyses.

COMPARATIVE CONSUMPTION OF COAL BY COMPOUND AND ORDINARY LOCOMOTIVES FOR THREE MONTHS ENDING 21ST MAY, 1886, ON THE GREAT EASTERN RAILWAY.

Number and Class of Engines.	1886. Four weeks ending.	Distance run.		Coal consumed.		
		Train Miles.	Engine Miles.	Total Cwts.	lbs. per Train Mile.	lbs. per Engine Mile.
Eleven compounds .	26 Mar.	36,503 $\frac{1}{2}$	37,794 $\frac{1}{2}$	9,849	30·2	29·1
Eleven „ .	23 Apl.	34,848 $\frac{1}{4}$	35,857 $\frac{1}{4}$	8,979	28·8	28·0
Eleven „ .	21 May	38,117 $\frac{3}{4}$	39,102 $\frac{3}{4}$	9,939	29·2	28·4
Totals and averages .	. .	109,469 $\frac{3}{4}$	112,754 $\frac{3}{4}$	28,767	29·4	28·5
Six ordinary . .	26 Mar.	18,610	19,355 $\frac{1}{2}$	5,724	34·4	33·0
Seven „ . .	23 Apl.	23,761	24,362 $\frac{1}{4}$	7,162	33·7	32·9
Seven „ . .	21 May	22,300	22,938 $\frac{3}{4}$	6,661	33·4	32·5
Totals and averages .	. .	64,671	66,656 $\frac{1}{4}$	19,547	33·8	32·8
Mean saving by compounds					4·4	4·3

COMPARATIVE CONSUMPTION OF COAL AND EVAPORATION OF WATER BY COMPOUND AND ORDINARY LOCOMOTIVES WORKING PASSENGER TRAINS FROM LONDON TO NORWICH ON THE GREAT EASTERN RAILWAY.

October, 1886.		Compound No. 704.	Ordinary No. 565.
Coal consumption, total .	lbs.	2780	3444
„ „ per mile .	lbs.	24·3	30·2
Water evaporation, total .	gallons	2196	2853
„ „ per lb. of coal .	lbs.	7·9	8·2
Feed water, average per 5 minutes .	gallons	112·5	126·4
Feed water temperature .	Fahr.	64°	65°
Average steam pressure per square inch .	lbs.	138	122
Load, London to Ipswich .	vehicles	14	15
„ Ipswich to Norwich .	vehicles	6	7

The compound steamed freely, weather very favourable.

The ordinary engine steamed moderately, weather rather unfavourable.

AVERAGE COMPOSITION OF COALS FROM DIFFERENT LOCALITIES.

(Phillips' Admiralty Coal Investigation.)

Locality.	Sp. Gr.	Carbon.	Hydrogen.	Nitrogen.	Sulphur.	Oxygen.	Ash.	Coke.
Average of 36 samples from Wales .	1·315	83·78	4·79	0·98	1·43	4·15	4·91	72·60
18 samples from New- castle	1·256	82·12	5·31	1·35	1·24	5·69	3·77	60·67
28 samples from Lan- cashire	1·273	77·90	5·32	1·30	1·44	9·53	4·88	60·22
8 samples from Scotland .	1·259	78·53	5·61	1·00	1·11	9·69	4·03	54·22
7 samples from Derby- shire	1·292	79·68	4·94	1·41	1·01	10·28	2·65	59·32

COMPOSITION OF VARIOUS ANTHRACITES.

Locality.	Sp. Gr.	Carbon.	Hydrogen.	Oxygen, Nitrogen, and Sulphur.	Ash.	Observers.
Pennsylvania . . .	1·462	89·21	2·43	3·69	4·67	} Regnault.
Swansea	1·348	91·29	2·33	4·80	1·58	
Mayenne	1·343	90·20	4·18	3·37	2·25	
Roldue (near Aix-la- Chapelle	1·367	90·72	3·92	4·42	0·94	
Swansea	1·270	90·58	3·60	4·10	1·72	} Jacquelin.
Sablé	1·750	87·22	2·49	3·39	6·90	
Vizille	1·730	94·09	1·85	2·85	1·90	
Iserre	1·650	94·00	1·49	3·58	4·00	

TABLE SHOWING THE PROGRESSIVE DIMINUTION OF HYDROGEN
AND OXYGEN FROM WOOD TO ANTHRACITE.*(Prof. Johnson.)*

	Carbon.	Hydrogen.	Oxygen.	Disposable Hydrogen.
Wood (average) . . .	100	12·18	83·07	1·80
Peat " . . .	100	9·85	55·67	2·89
Lignite (mean of 15 varieties) . . .	100	8·37	42·42	3·07
Coal, South Staffordshire . . .	100	6·12	21·23	3·47
Steam coal, Newcastle . . .	100	5·91	18·32	3·62
Portrefelin coal, S. Wales . . .	100	4·75	5·28	4·09
Pennsylvanian anthracite . . .	100	2·84	1·74	2·63

ANALYSIS OF PATENT FUELS.

(Admiralty Investigation.)

Kind of Fuel.	Sp. Gr.	Carbon.	Hydrogen.	Nitrogen.	Sulphur.	Oxygen.	Ash.	Coke.
Warlich's patent fuel . . .	1·15	90·02	5·56	trace	1·62	—	2·91	85·1
Livingstone's steam fuel . . .	1·184	86·07	4·13	1·80	1·45	2·03	4·52	—
Lyon's patent fuel . . .	1·13	86·36	4·56	1·06	1·29	2·07	4·66	—
Wylam's " . . .	1·10	79·91	5·69	1·68	1·25	6·63	4·84	65·8
Bell's " . . .	1·14	87·88	5·22	0·81	0·71	0·42	4·96	71·7
Holland & Green's patent fuel . . .	1·302	70·14	4·65	1·15	—	—	13·73	—

AVERAGE VALUE OF COALS FROM DIFFERENT LOCALITIES.

Locality.	Lbs. of water evaporated from 100° C. by 1 lb. of coal.	Number of lbs. evaporated per hour.	Weight in lbs. of 1 cubic foot of coal as used for fuel.	Space occupied by 1 ton in cubic feet.	Results obtained in experiments on cohesive power of coals (percentage of large coals).	Per cent. of sulphur in coals.
Average of 37 samples from Wales	9.05	448.2	53.1	42.71	60.9	1.42
Average of 17 samples from Newcastle	8.37	411.1	49.8	45.30	67.5	0.94
Average of 28 samples from Lancashire	7.94	447.6	49.7	45.15	73.5	1.42
Average of 8 samples from Scotland	7.70	431.4	50.0	49.99	73.4	1.45
Average of 8 samples from Derbyshire	7.58	432.7	47.2	47.45	80.9	1.01

COMPARATIVE VALUES OF WELSH STEAM COALS.

(Portsmouth Dockyard Experiments.)

	lbs. of water evaporated by 1 lb. of coal.	Percentage of clinker and ash.
Nixon's Navigation	10.05	5.37
Wayne's Merthyr	10.05	5.37
Thomas	9.79	5.47
Nauhudyn "	9.62	5.48
Ynsfaio	9.52	6.76
Merthyr Dare	9.45	5.48
Resolven Merthyr	9.41	6.04
Insoles	9.37	6.52
Averages	9.65	5.81

**RESULTS OF EVAPORATIVE DUTIES OF NEWCASTLE AND WELSH
COALS IN THE COAL-TESTING MARINE BOILER AT KEYHAM STEAM
FACTORY.**

Coal.	Area of Fire-grate.	Coal consumed per hour.	Coal per square foot of grate, per hour.	Water consumed from 100° per hour.	Water per sq. foot of grate per hour.	Water evaporated from 100° C. per lb. of coal.
FIRST SERIES.	sq. ft.	cwts.	lbs.	cub. ft.	cub. ft.	lbs.
(With common doors.)						
Welsh, Wayne's Merthyr, Resolven, Merthyr Dare, Gellia, Cadoxton . . .	14'0	1'93	15'44	32'4	2'31	10'42
Hartley Main Newcastle . . .	14'0	2'32	18'56	34'5	2'46	9'22
$\frac{1}{2}$ Welsh $\frac{1}{2}$ Hartley . . .	14'0	1'92	15'40	30'4	2'17	9'81
2 " 1 " . . .	14'0	1'76	14'08	28'7	2'05	10'12
1 " 2 " . . .	14'0	1'96	15'70	30'7	2'20	9'72
(With perforated doors.)						
Hartley Main . . .	14'0	2'06	16'50	30'2	2'16	9'10
SECOND SERIES.						
(With common doors.)						
Welsh—Powell's Duffryn, Nixon's Navigation, Davis's Merthyr . . .	14'0	2'09	16'68	37'1	2'65	11'05
Newcastle — Davidson's Hartley, Hasting's Hartley . . .	14'0	2'29	18'29	34'5	2'46	9'39
$\frac{1}{2}$ Welsh $\frac{1}{2}$ Hartleys . . .	14'0	2'03	16'24	34'4	2'46	10'56
2 " 1 " . . .	14'0	2'43	16'36	35'0	2'50	10'61
Welsh . . .	14'0	2'19	17'48	39'3	2'80	11'16
THIRD SERIES.						
(With perforated doors.)						
Welsh coal . . .	14'0	1'87	14'95	32'7	2'34	10'86
Hartleys . . .	14'0	2'13	17'04	32'8	2'34	9'61
$\frac{1}{2}$ Welsh $\frac{1}{2}$ Hartleys . . .	14'0	2'18	17'44	37'1	2'65	10'54
2 " 1 " . . .	14'0	2'08	16'64	35'7	2'55	10'64
1 " 2 " . . .	14'0	2'18	17'42	36'5	2'61	10'39
Davidson's Hartley . . .	14'0	2'86	22'88	42'9	3'06	9'31
$\frac{1}{2}$ Hartley $\frac{1}{2}$ Welsh . . .	14'0	2'30	18'40	31'0	2'22	10'80
FOURTH SERIES.						
(With smaller grate area. With common doors.)						
Welsh coal . . .	10'5	2'11	22'46	38'3	3'65	11'31
$\frac{1}{2}$ Welsh small $\frac{1}{2}$ Davidson's Hartley . . .	10'5	2'02	21'60	36'0	3'43	11'06
$\frac{1}{2}$ Welsh beans $\frac{1}{2}$ Hasting's Hartley . . .	10'5	2'14	22'85	36'7	3'50	10'65
FIFTH SERIES.						
(With perforated doors.)						
Hartleys . . .	10'5	2'29	24'40	42'0	4'00	11'42
$\frac{1}{2}$ Welsh $\frac{1}{2}$ Davidson's Hartley . . .	10'5	2'10	22'34	39'3	3'74	11'65

DISPOSAL OF HEAT OF COMBUSTION OF COAL BURNT UNDER STEAM BOILERS, AND COMPOSITION OF THE PRODUCTS OF COMBUSTION.

The following are results obtained by MM. Scheurer-Kestner and Meunier in 1868 in a French boiler at Thann. (*Vide Bulletin de la Société Industrielle de Mulhouse.*)

	sq. ft.
Heating surface of heaters	301·3
„ „ boiler	129·1
Total of boiler	430·4
Heating surface of feed heaters	764·0
	<hr/>
	1,194·4
Direct heating surface exposed to fire	32·3
Area of fire-grate	19·3
Area of air-spaces through grate	5·5
Ratio of grate area to total surface of boiler	1 to 22·3
Ratio of grate area to total surface of boiler and fuel heaters	1 to 63

The average composition of the Ronchamp coal used in the experiments devoted to the analysis of the chimney gases was as follows :—

Carbon	70
Hydrogen	4
Oxygen	4
Nitrogen	1
Ash	21
	<hr/>
	100

ANALYSIS OF THE PRODUCT OF COMBUSTION OF RONCHAMP
COAL UNDER A FRENCH BOILER AT THANN.

No. of Experiments.	Coal per sq. ft. grate per hour.	Weight of each Charge.	Intervals of Charges.	Composition of the Gases.						Total Air per lb. of Coal.
				Carbonic Acid.	Carbonic Oxide.	Carbon Vapour.	Hydrogen.	Nitrogen.	Free r.	
	lbs.	lbs.	mins.	p. c.	p. c.	p. c.	p. c.	p. c.	p. c.	cu. ft.
12	8.2	15.4	5	14.9	.84	1.15	1.35	75.1	6.7	110.0
11	9.6	30.8	8	14.2	.97	1.11	1.11	72.5	10.5	116.2
9	9.6	15.4	4	14.6	.86	.56	.56	70.1	13.3	134.7
14	8.2	30.8	10	13.4	.24	1.41	1.41	63.7	20.9	144.5
13	8.2	15.4	5	13.3	—	.91	.91	67.7	17.6	147.5
8	4.7	15.4	8	12.9	—	.96	.96	59.7	26.2	156.6
10	19.0	15.4	2	10.9	—	.19	.19	45.9	42.8	198.3
7	3.4	13.2	10	8.2	—	0.4	.52	37.4	53.8	260.5

CARBON AND HYDROGEN IN ESCAPED COMBUSTIBLE GASES.

No. of Experiments.	Per cent. of Total Carbon.			Per cent. of Total Hydrogen.	Temperature of Gases leaving Feed heaters.
	In Oxide.	In Hydrocarbons.	Total.		
12	4.1	11.4	15.6	19.5	119° C.
11	5.0	10.2	15.3	16.7	128
9	5.2	5.9	11.2	9.9	126
14	1.5	4.1	5.7	26.9	—
13	—	—	6.1	17.5	135
8	—	—	3.9	19.7	93
10	—	—	3.4	4.7	156
7	—	—	.9	17.7	94

It appears from these results that the most effective combination is arrived at when about one-third of the gaseous products consists of free air.

Influence of Excess of Air.—The following table shows the influence of excess of air in using Friedrichsthal and Altenwald coal:—

Friedrichsthal Coal.		Altenwald Coal.	
Free Air. Per cent.	Lbs. Water Evap. per lb. of Coal.	Free Air. Per cent.	Lbs. Water Evap. per lb. of Coal.
40	6.80	35	7.06
36	6.46	33	7.28
30	6.38	32	7.02
27	6.19	30	6.79
27	6.23	28	6.85
24	5.68	25	6.71
23	5.80	23	6.66

Distribution of the Heat of Combustion.—For the comparison of the absolute heat of combustion of coals, the boiler, firegrate, &c., in this case had the following dimensions:—

	sq. ft.
Area of fire-grate 4.6 feet long by 4.47 feet wide . . .	20.6
Ratio of grate to heating surface of boiler . . .	1 to 21
„ to total service of boiler and feed heaters . . .	1 to 5
	cu. ft.
Total capacity of boiler	423.6
„ „ feed heaters	317.7
Water room in boiler	335.3
Steam room in boiler :	88.3
	sq. ft.
Heated surface of brickwork for conduction and radiation of heat above	1,290

The following are the results obtained with various coals in the above boiler:—

Coal.	Observed Total Heat of Combustion. Units.	Air at 1° C. per lb. of Coal. Cu. ft.	Free Air. Per cent.	Temperatures.			Ash.	Pressure of Steams. Atmosphere.	Water per lb. of Coal from and at 100° C.
				Air. °C.	Feed Heater. °C.	Smoke. °C.			
Ronchamp, No. 3 .	7,825	152·3	24·7	17	65	132	17·3	4·46	8·77
„ No. 4 .	7,775	160·7	29·1	21	71	138	15·8	4·84	9·49
Sarrebrück (mean of 7 coals)	7,500	159·0	31·0	20	72	130	14·0	4·72	8·17
Blanzy, Montceau .	7,067	135·4	23·9	18	65	160	12·0	4·60	7·89
„ anthracite .	7,125	152·3	30·5	16	64	175	24·4	4·58	8·18
Creusot, anthracite .	8,949	269·0	47·6	—	72	144	9·1	4·60	10·53
Creusot, $\frac{2}{3}$; Ronchamp, $\frac{1}{3}$	8,565	230·1	36·2	11	67	132	13·4	4·71	10·54
Creusot, $\frac{2}{3}$; Ronchamp, $\frac{1}{3}$	8,630	214·9	34·2	8	62	145	15·9	4·71	9·83
Wood, charcoal . .	8,080	250·4	42·5	—	—	155	0·5	—	9·2

The absolute heat of combustion of the fuels was estimated from the above to have been distributed about as follows:—

	per cent.
Heat in the steam (about 60 lbs. pressure)	61·0
Heat ungenerated in the combustible gases	5·5
Heat lost in the clinker and ash	1·5
Heat carried off in the gaseous products of combustion .	5·5
Heat ungenerated in the smoke carbon	·5
Heat absorbed in the evaporation of the hygrometric water, and water newly formed	2·5
Heat lost in the brickwork	23·5
Total	100·0

HOLDEN'S SYSTEM OF USING LIQUID FUEL ON LOCOMOTIVE AND STATIONARY BOILERS.

By this system liquid fuel and air are introduced into the fire-box above a thin layer of solid incandescent fuel by means of a special injector, Fig. 16, and burnt in combination with the solid fuel, without any alteration of the fire-box, other than the insertion of one or more tubes through the casings, the boiler so fitted being equally suitable

for the use of ordinary fuel. The internal arrangement of the injector varies, according as the appliance is to be used with

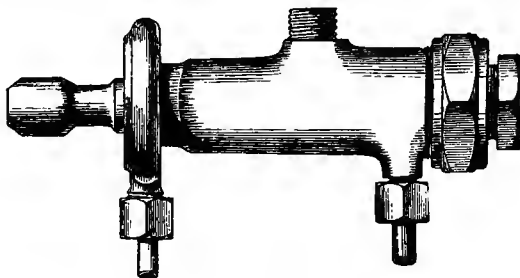


FIG. 16.

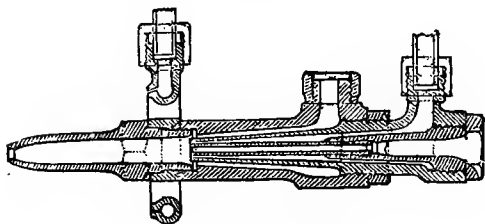


FIG. 17.—Stationary Pattern.

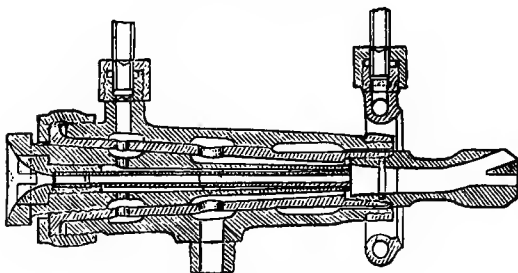


FIG. 18.—Locomotive Pattern.

HOLDEN'S LIQUID FUEL INJECTOR.

the boiler of a stationary engine, as in Fig. 17, or with the boiler of a locomotive engine, as in Fig. 18.

The use of this method results in effectual combustion, entire

absence of smoke, intense and regular heat, and great economy of fuel. Slack, inferior coal, lignite, cinders, wood, peat, or sawdust may with equally good results be used as solid fuel. The air necessary for combustion, not having to be introduced through the fire, a very small amount of draught is required, and in the case of locomotives the orifice of the blast pipe may advantageously be enlarged from 50 to 60 per cent., thus reducing the wear and tear of the fire-box, tubes, smoke-box, and chimney, preventing the emission of sparks and ashes, and conducing to economic and efficient working by the diminution of back pressure.

The Steam Pressure in a boiler fitted with the apparatus can be regulated with great nicety, as the solid fuel required is only sufficient to form a base for ignition, and the steam pressure can be promptly increased or reduced by varying the quantity of liquid fuel. For locomotives this is specially advantageous, as in the case of an exceptionally heavy load, strong wind, adverse gradient, or an engine employed piloting or shunting, a good supply of steam can be quickly ensured and easily maintained. On the other hand, if an engine is suddenly stopped by signals, or has to put by for other trains to pass, the generation of steam can be immediately stopped.

In the event of tubes or stays leaking, or an engine having to face a bank towards the end of a journey when the fire is dirty, steam can be maintained and the train worked with the help of liquid fuel, when without it it would probably lose time and come to a stand.

It is therefore submitted that even in cases where the relative cost of coal and liquid fuel is in favour of coal burning, the advantages to be derived from the system are sufficient to render its adoption desirable as a supplementary means of meeting any exceptional demands upon the engine.

RESULTS OBTAINED WITH LIQUID FUEL BY MR. JAMES HOLDEN,
M.I.C.E., ON THE GREAT EASTERN RAILWAY.
EXPERIMENT ON A SMALL CORNISH BOILER.

Coal only used.

1887. Consumption during one week from August 15th to

20th (inclusive), $74\frac{1}{4}$ hours' work, including lighting up = $80\frac{1}{2}$ cwt. = 121·3 lbs. per hour.

Cost for 100 hours = 12,130 lbs. of coal at 11s. per ton = £2 19s. $7\frac{1}{2}$ d.

Coal, Coke, and Tar used. Holden's System.

1888. Consumption during one week from June 25th to 30th (inclusive), $87\frac{3}{4}$ hours working; including lighting up

= coal 15 cwt. = 19·2 lbs. per hour

= coke $11\frac{1}{2}$ cwt. = 14·7 „ „

Gas tar 280 gallons = 35·1 „ „

Total 69·0

Cost for 100 hours

= 1,920 lbs. of coal at 11s. per ton = 0 9 $5\frac{1}{2}$

= 1,470 lbs. of coke at 9s. 6d. „ = 0 6 $1\frac{1}{2}$

= 3,510 lbs. of tar at 12s. 6d. „ = 0 19 $7\frac{1}{2}$

Total £1 15 $2\frac{1}{2}$

COMPARATIVE COST OF LIQUID FUEL AND COAL ON LOCOMOTIVES OF THE GREAT EASTERN RAILWAY.

No. of Engine.	Total Miles run.	Total lbs. used.			Lbs. per Mile.			Total lbs. of Coal, Liquid Fuel, and Chalk per mile.	Proportion of Liquid Fuel and Chalk to Coal used.	Total cost of Fuel.	Cost per mile in Pence.
		Coal.	Liquid Fuel.	Chalk.	Coal.	Liquid Fuel.	Chalk.				
193 951 $\frac{1}{2}$	13,511	10,505	784	14·2	11·0	8	26·0	83	per cent.	£ s. d.	
194 951 $\frac{1}{2}$	27,738	—	—	29·1	—	—	29·1	—	—	9 1 5	2·28
										9 4 10 $\frac{1}{2}$	2·33
Difference in favour of No. 193 engine . . .										0 3 5 $\frac{1}{2}$	·05

N.B. Cost of coal computed at 14/11 per ton (Radford coal used).

„ liquid fuel computed at 1 $\frac{1}{8}$ d. per gallon of 11 lbs.

„ chalk computed at 5/6 per ton.

COMPARATIVE EVAPORATION OF WATER BY DIFFERENT LIQUID FUELS INJECTED AND SPRAYED WITH HOLDEN'S PATENT INJECTOR.

ON A SMALL VERTICAL BOILER ABOUT 6-H.P. WORKING AT 50 LBS. PER SQUARE INCH PRESSURE.					Evaporative power of Liquid Fuel alone.
With Yorks coal		4.7 lbs. water per lb. of fuel			
" "	and coal tar	5.7	" "	" "	6 lbs.
" "	and green oil	6.6	" "	" "	7.3 "
" "	and astatki	9.1	" "	" "	10.2 "
ON A SMALL CORNISH BOILER ABOUT 30-H.P. WORKING AT A PRESSURE OF 35 LBS. PER SQUARE INCH.					
With Yorks coal		8.4 lbs. water per lb. of fuel			
" "	and coal tar	9.9	" "	" "	11.3 lbs.
" "	and green oil	10.3	" "	" "	12.3 "
" "	and astatki	12.3	" "	" "	14.5 "
ON A LOCO. BOILER WORKING AS STATIONARY AT 80 LBS. PER SQUARE INCH PRESSURE.					
With Yorks coal		9.1 lbs. water per lb. of fuel			
" "	and green oil	12.9	" "	" "	15.1 lbs.

In all cases the figures give evaporation from feed water at atmospheric temperature.

Shale oil about equals green oil for evaporative value.

COMPARATIVE CONSUMPTION OF COAL AND OIL IN ORDINARY AND COMPOUND PASSENGER LOCOMOTIVES ON THE BUENOS AYRES AND ROSARIO RAILWAY.

Four weeks from 27 May to 25 June, 1887.	Distance run.	Coal, lbs.		Oil, lbs.	
		Total.	Per Mile.	Total.	Per 100 Miles.
Ordinary engine, No. 10.	3,937	111,001	28.19	595.24	15.12
Compound " No. 34.	3,638	81,350	22.36	599.65	16.48

Thus the compound burnt $20\frac{1}{2}$ per cent. less coal than the

ordinary engine, but used 9 per cent. more oil; the actual money saving was 3 dollars 20 cents per hundred miles. The working pressure in the ordinary engine was 150 lbs. per square inch, and in the compounds 160 lbs.

The following are very instructive results obtained by Mr. Urquhart on the Grazi and Tsaritsin Railway locomotives, showing the efficiency of petroleum refuse:—

COMPARATIVE TRIALS WITH PETROLEUM, ANTHRACITE, BITUMINOUS COAL, AND WOOD, BETWEEN ARCHEDA AND TSARITSIN ON GRAZI AND TSARITSIN RAILWAY IN WINTER.

Train alone.		Distance run.	Car. Miles.	Fuel.	Consumption, including Lighting-up.		Cost of Fuel per Train Mile.	Atmospheric Temperature and Weather.
No. of Loaded Cars.	Gross Load.				Total.	Per Train Mile.		
25	tons. 400	miles. 388	9,700	Anthracite	lbs. 31,779	lbs. 81.90	pence. 11.957	—14° to —13° C. Strong side wind.
25	400	388	9,700	{ Bituminous coal }	37,557.5	96.53	14.093	
25	400	194	4,850	{ Petroleum refuse }	9,462	48.77	5.487	
25	400	194	4,850	Anthracite	12,639.5	65.15	9.512	—6° to —11° C. Light side wind.
25	400	194	4,850	{ Wood in billets }	Cubic ft. 1,071.8	Cu. ft. 5.52	8.5	
25	400	194	4,850	{ Petroleum refuse }	lbs. 7,223	lbs. 37.23	4.188	

Prices of Fuel:—Petroleum refuse, 21s. per ton.

Anthracite and bituminous coal, 27s. 3d. per ton.

Wood, 1.47d. per cubic foot.

Dimensions of Locomotives:—Cylinders, 18½" dia. and 24" stroke. Wheels, 4 ft. 3 in. dia. Total heating surface, 1,248 sq. ft. Total adhesion weight, 36 tons. Boiler pressure, 8 to 9 atmos.

COMPARATIVE MONTHLY AVERAGES DURING 1883 WITH COAL AND PETROLEUM REFUSE IN LOCOMOTIVES, WORKING
MAIN LINE TRAINS ON GRAZI AND TSARITSIN RAILWAY.

CONSUMPTION OF FUEL PER TRAIN MILE.															
Locomotives.	Trains.	Fuel.	Monthly Averages of Consumption per Train Mile in lbs.												Mean.
			Jan.	Feb.	Mar.	April.	May.	June.	July.	Aug.	Sept.	Oct.	Nov.	Dec.	
Eight wheels coupled	Goods	Coal	98'06	108'96	100'79	76'27	76'27	79'00	74'91	73'55	79'00	85'81	98'06	95'34	87'17
Six wheels coupled	Goods	Coal and Petroleum refuse	73'55	77'63	70'82	64'01	55'84	61'29	54'48	55'84	65'38	80'36	92'62	85'81	69'80
Four wheels coupled	Mixed	Coal	53'12	54'48	46'31	42'22	34'05	35'41	31'33	36'10	40'86	39'50	50'39	54'48	43'19
Four wheels coupled	Passenger	Coal and Petroleum refuse	51'76	76'27	43'58	34'05	36'77	35'41	42'22	49'03	51'76	40'86	49'93	58'57	47'44
			40'86	49'03	46'31	36'77	34'05	32'69	31'33	32'69	36'77	39'50	42'22	50'39	39'38
			—	—	—	—	—	—	—	—	20'43	31'33	32'69	34'05	29'62
COST OF FUEL PER TRAIN MILE.															
Locomotives.	Trains.	Fuel.	Monthly Averages of Cost per Train Mile in Pence.												Mean.
			Jan.	Feb.	Mar.	April.	May.	June.	July.	Aug.	Sept.	Oct.	Nov.	Dec.	
Eight wheels coupled	Goods	Coal	13'495	15'635	14'346	11'226	11'319	11'704	11'186	10'925	11'552	12'471	14'600	13'930	12'699
Six wheels coupled	Goods	Coal	10'520	11'099	9'897	9'405	8'387	9'317	8'217	8'279	9'694	11'776	13'647	12'344	10'212
Four wheels coupled	Mixed	Petroleum refuse	7'294	7'602	6'158	4'973	4'040	4'163	3'617	4'170	4'948	4'771	6'817	7'356	5'495
Four wheels coupled	Passenger	Coal	7'353	10'973	6'672	4'934	5'466	4'988	6'267	7'269	11'215	5'908	7'102	8'652	6'932
			6'070	7'023	6'602	5'144	4'829	4'670	4'445	4'819	5'165	5'633	6'111	7'580	5'672
			—	—	—	—	—	—	—	—	2'411	5'611	4'366	4'634	3'808

Of the Coal consumed 49 per cent. was anthracite and 51 per cent. was bituminous coal.
Mean Price of Fuel :—Petroleum refuse, 23s. 4d. per ton; Coal, 27s. per ton.

COMPARATIVE VALUE OF LIQUID FUEL AND COAL. 141

The following results were obtained on the above line in summer time :—

Train alone.		Train Miles.	Fuel.	Consumption, including Lighting-up.		Cost of Fuel per Train Mile.
No. of Loaded Cars.	Gross Load.			Total.	Per Train Mile.	
No.	Tons.			lbs.	lbs.	pece.
30	480	194	Bituminous coal	14,084·07	72·598	10·599
30	480	194	Petroleum refuse	6,175·325	31·831	3·581
30	480	194	Anthracite	12,784·002	65·897	9·621
30	480	194	Petroleum refuse	6,103·097	31·459	3·539

Theoretical Evaporative Value of Petroleum Fuel and Coal.

Fuel.	Specific Gravity at 32° F.	Chemical Composition.				Heating Power. British Thermal Units.	Theoretical Evaporation.	
		Carbon, per Cent.	Hydrogen, per Cent.	Oxygen, per Cent.	Sulphur, per Cent.		Lbs. of Water per lbs. of Fuel.	
							From and at 212° F.	At 8½ Atmo- spheric effective Pressure.
Pennsylvanian heavy crude oil	·886	84·9	13·7	1·4	—	20,736	lbs. 21·48	lbs. 17·8
Caucasian light crude oil	·884	86·3	13·6	0·1	—	22,027	22·79	18·9
Caucasian heavy crude oil	·938	86·6	12·3	1·1	—	20,138	20·85	17·3
Petroleum refuse	·928	87·1	11·7	1·2	—	19,832	20·53	17·1
Good English coal, mean of 98 sam- ples	1·380	80·0	5·0	8·0	1·25	14,112	14·61	12·16

In comparing petroleum refuse (Russian) and anthracite, Mr. Urquhart says that " the former has a theoretical evapora-

tive power of 16.2 lbs. of water per lb. of fuel, and the latter of 12.2 lbs., at an effective pressure of 8 atmospheres, or 120 lbs. per square inch; hence petroleum has, weight for weight, 33 per cent. higher evaporative value than anthracite. Now in locomotive practice, a mean evaporation of from 7 to $7\frac{1}{2}$ lbs. of water per lb. of anthracite, is about what is generally obtained; thus giving about 60 per cent. of efficiency, while 40 per cent. of the heating power is unavoidably lost. But with petroleum an evaporation of 12.25 lbs. is practically obtained, giving $\frac{12.25}{16.2} = 75$ per cent. efficiency. Thus, in the first place, petroleum is theoretically 33 per cent. superior to anthracite in evaporative power, and, secondly, its useful effect is 15 per cent. greater, being 75 per cent. instead of 60 per cent.; while, thirdly, weight for weight, the practical evaporative value of petroleum must be reckoned as at least from $\frac{12.25 - 7.50}{7.50} = 63$ per cent. to $\frac{12.25 - 7.00}{7.00} = 75$ per cent. higher than that of anthracite.

Analyses of Various Gaseous Fuels.

ANALYSIS OF SIEMENS PRODUCER GAS.

(*Trans. Amer. Inst. Min. Eng.*)

	1	2	3	4	5
	per cent.	per cent.	per cent.	per cent.	per cent.
Carbonic acid . . .	3.9	8.6	9.3	1.5	6.1
Carbonic oxide . . .	27.3	20.0	16.5	23.6	22.3
Hydrogen	—	8.7	8.6	6.0	28.7
Marsh gas	1.4	1.2	2.7	3.0	1.0
Nitrogen	67.4	61.4	62.9	65.9	41.9
Heat units	93966	97184	99074	114939	164164

ANALYSIS OF AMERICAN NATURAL GAS.

(Ford, Jr. I. and S. Inst.)

	1	2	3	4	5
	per cent.	per cent.	per cent.	per cent.	per cent.
Carbonic acid . . .	0·8	0·6	nil.	0·4	nil.
Carbonic oxide . . .	1·0	0·8	0·58	0·4	1·00
Oxygen	1·1	0·8	0·78	0·8	2·10
Olefiant gas	0·7	0·8	0·98	0·6	0·80
Ethylic hydride . . .	3·6	5·5	7·92	12·3	5·20
Marsh gas	72·18	65·25	60·70	49·58	57·85
Hydrogen	20·02	26·16	29·03	35·92	9·64
Nitrogen	nil.	nil.	nil.	nil.	23·41
Heat units	728746	698852	627170	745813	592380

ANALYSIS OF THE GASES AT DIFFERENT DEPTHS OF THE ALFRETON BLAST FURNACE.

	Distance below the Furnace Mouth.				
	8 feet	14 feet	20 feet	24 feet	34 feet
Nitrogen	54·77	50·95	60·46	56·75	58·05
Carbonic acid	9·42	9·10	10·83	10·08	—
Carbonic oxide	20·24	19·32	19·48	25·19	37·43
Marsh gas	8·23	6·64	4·40	2·33	—
Hydrogen	6·49	12·42	4·83	5·65	3·18
Olefiant gas	0·85	1·57	—	—	—
Cyanogen	—	—	—	trace	1·34
	100·00	100·00	100·00	100·00	100·00

ANALYSIS OF WATER-GAS.

	(1) "Strong" Gas (Dr. Moore).	(2) Water-Gas Coke used (Langlois).	(3) Water-Gas Coke used (Frankland).	(4) Gas made by Beilby's Process.
Carbonic acid	2·05	12·000	13·80	21·32
Carbonic oxide	35·88	31·860	29·30	10·72
Marsh gas	4·11	1·62	56·9	nil.
Hydrogen	52·76	54·52		
Oxygen	0·77	—	—	—
Nitrogen	4·43	—	—	30·77
	100·00	100·00	100·00	100·00

ANALYSIS OF GASES ISSUING FROM THE MOUTH OF THE
BESSEMER CONVERTER.*(Snelus, I. and S. Inst.)*

	Time after Commencement of Blow.						After addition of Spiegeleisen at Bochum Works.	
	2 mins.	4 mins.	6 mins.	10 mins.	12 mins.	14 mins.		
Carbonic acid . . .	10·71	8·59	8·20	3·58	2·30	1·34	—	0·86
Carbonic oxide . . .	nil.	3·95	4·52	19·59	29·30	31·11	82·6	78·55
Oxygen	0·92	—	—	—	—	—	—	1·32
Hydrogen	88·37	0·88	2·00	2·00	2·16	2·00	2·8	2·52
Nitrogen		86·58	85·28	74·83	66·24	65·55	14·3	16·38

COMPOSITION OF GASES IN BLOWHOLES OF STEEL INGOTS.

(Stead, Clev. Inst. Eng.)

	Steel containing C = 0·42 p. c. Si = 1·00 p. c. Mn = 1·08 p. c.	Steel containing C = 0·33 p. c. Si = 0·10 p. c. Mn. = 0·69 p. c.	Steel containing C = 0·17 p. c. Si = 0·09 p. c. Mn. = 0·89 p. c.
Hydrogen	67·10	86·62	87·21
Nitrogen	30·30	13·29	11·15
Carbonic oxide	2·60	0·32	1·64
Oxygen	—	0·37	—

COMPOSITION OF VARIOUS GASES.

(By Weight.)

	N	H	CO ₂	CO	CH ₄	C ₂ H ₄	Autho- rity.
Blast furnace, Scotch . .	48·20	0·90	21·70	29·24	—	—	Bell
Blast furnace, Askam . .	52·59	0·14	13·47	33·80	—	—	Crossley
Blast furnace, Cleveland .	58·54	0·06	14·32	27·03	—	—	Stead
Producer gas, Siemens . .	64·50	—	6·95	24·92	0·89	2·73	Snelus
Producer gas, Siemens . .	63·22	0·65	8·71	25·97	1·45	—	"
Producer gas, Wilson . . .	61·70	0·90	6·91	29·58	0·91	—	Stead
Producer gas, Wilson . . .	62·84	1·11	8·29	26·33	1·43	—	"
Retort-made town gas . . .	—	8·17	—	18·61	56·10	17·12	—

AVERAGE COMPOSITION OF COAL GAS.

(Thorpe.)

Hydrogen	45·58
Methane	34·90
Carbon Monoxide	6·64
Ethene	4·08
Quartene	2·38
Sulphuretted hydrogen	0·29
Nitrogen	2·46
Carbonic acid	3·67
	<hr/> 100·00

Variable Blast Pipes on Locomotives.—An important economiser of fuel on locomotives is the somewhat recent invention by Mr. Macallan, M.I.M.E., of a “variable blast pipe.” The appliance is fully under the control of the driver, who can at will regulate an appropriate influx of air, by increasing or decreasing, as the case may demand, the diameter of the nozzle of the blast pipe, by means of the adjustable cap, A, shown in Fig. 19, which is manipulated from the foot-plate by a rod connected with the lever B. It is claimed that on an average a saving of 10 per cent. of fuel is effected by its means. The reason of this is not far to seek. The ordinary blast pipe of locomotives has an area such as will cause a sufficiently strong blast to enable the engine to make steam to do its heaviest work, while when doing light work the exhaust steam passes through the same orifice, and consequently causes more fuel to be burnt than is required to raise the required amount of steam.

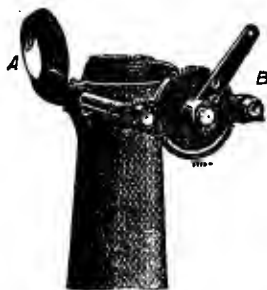


FIG. 19.—MACALLAN'S VARIABLE BLAST PIPE.

PART IV.

WATER.

It is intended in this section to deal with waters required to be used (1) for boilers, and (2) for drinking purposes.

I.—WATER FOR BOILER PURPOSES.

Waters largely impregnated with carbonates and sulphates of lime and magnesia, are highly objectionable for use in steam boilers, since these salts are deposited as a hard incrustation on the boiler, tubes, &c., causing great inconvenience and waste of fuel, and any considerable quantities of such salts, as chloride, sulphate and carbonate of soda, &c., present in waters, although not causing "hardness," give much trouble in the form of "priming." Waters containing a comparatively large proportion of calcareous and magnesian salts are termed "Hard," while those containing a small proportion are termed "Soft." These qualities are practically determined by the more or less "curd" (lime and magnesian soaps) that is produced, when the water is used with soap. The amount of mineral matter or incrustating substances present in a water, materially depends upon the geological character of the strata through which the water passes or percolates. The following table shows the quality of the water obtained from the various geological strata or formations of this country :—

Formations.	Thickness in Feet.	Quality of Water.
Chalk	645 to 1,000	Hard.
Upper Greensand	100 to 400	Rather Hard.
Lower Greensand	20 to 500	Soft and Good.
Purbeck and Portland Beds	0 to 60	Rather Hard.
Coral Rag and Grit	40	"Hard."
Great and Inferior Oolites	200 to 450	Soft.
Upper Lias Sands	20 to 200	Rather Hard.
Marlstone or Middle Lias	30 to 250	Soft or Variable.
New Red Sandstone	0 to 2,150	Soft.
Lower Permian Beds	Variable.	

Collection of Samples.—For collecting the water, the stoppered glass bottles known as "Winchester quarts," holding $2\frac{1}{2}$ litres, are generally found convenient. One of these filled will be sufficient for an ordinary analysis, but when an exhaustive examination is proposed at least three will be required.

If the water is to be taken from a pump or tap, let a few gallons of the water be run off before collecting; then fill the bottle to the top to expel any gases, empty, wash out the bottle about half a dozen times with the water, and then fill to within about an inch from the stopper, and tie down.

When the water is obtained from a well, tank, or river, the clean bottle is plunged into the water, and the neck kept below the surface, care being taken that no scum, &c., gets into the bottle, and that no mud or sediment is disturbed.

When the water is intended to be examined as to its suitability for dietetic purposes, it should be kept in a cool, dark place previous to its analysis, which should take place within forty-eight hours after its collection, since on long keeping, any organic matter may be decomposed by fermentation, &c.

In judging of the suitability of a water for boilers, &c., the following points must be determined :—(1) Mechanically suspended matter; (2) Total solid matter in solution; (3) Hardness—total, permanent, and temporary; (4) Chlorine in chlorides; (5) Alkalinity; and (6) Qualitative idea of the amounts of sulphates, nitrates, lime, and magnesia present.

This will give a tolerably good idea of the nature and suitability of water for use in steam boilers, although sometimes a complete mineral analysis of a water is desirable.

Determination of Suspended Matter.—If on shaking up the sample of water a fair quantity of suspended matter is seen, filter off about 250 cc. of the sample through a weighed filter, wash with distilled water, dry at 130° C., cool and weigh. Increase of weight multiplied by 280 = grs. suspended matter per gallon.

Total Solid Matter in Solution.—Accurately weigh a porcelain or platinum dish of about 100 cc. capacity, and measure out 70 cc. of the filtered sample from a pipette into it. Place on a water bath, evaporate to dryness, remove to an air bath regulated to 130° C. for one hour, transfer to a desiccator, allow to cool, and quickly weigh. Place in the air bath for another hour at 130° C., cool and weigh as before. If the second weight is no less than that of the previous one, the increase in weight over the dish = total solid matter in 70 cc., and which multiplied by 1,000 gives grains per gallon of solid matter.

Determination of Hardness.—The degree of hardness of a water is determined by ascertaining the amount of *standard* soap solution necessary to form a permanent lather with a definite volume of the sample: the “harder” the water the more soap will it consume, owing to the formation of insoluble calcium, magnesium, &c., soaps (“curd”), brought about by the decomposition of the soda or potash soap added, by the salts of the alkaline earths present in the water. The hardness of a water is usually expressed in terms of carbonate of lime, or degrees Clark (*i.e.*, grains per gallon), Dr. Clark being the originator of the process; and is usually determined in terms of “temporary” and “permanent” hardness. By “temporary” is meant that hardness which can be eliminated by boiling the water, by which means the carbonic acid gas which

holds the carbonates of lime and magnesia, &c., in solution is expelled, and the carbonates in consequence precipitated. If this boiled water be filtered, the hardness ("permanent") of the filtrate will be diminished in proportion to the amounts of carbonates filtered off. Carbonate of lime is not, however, completely precipitated by boiling, 2.5 grains per gallon being soluble in water free from carbonic acid. In order to ascertain the hardness, a *standard* "hard" water and a *standard* soap solution will be required.

Preparation of Standard "Hard Water."—Dissolve 1.11 grms. of pure fused calcium chloride in a little water, and dilute to 1,000 cc. at 15° C., or dissolve 1 gram. of pure carbonate of lime in 50 cc. of 5 E HCl, evaporate to dryness, dissolve in 50 cc. of water, and carefully neutralize any trace of free acid with 5 E AmHO. In either case each cc. of the solution will correspond, or be chemically equivalent, to .001 gram. of carbonate of lime.

Preparation of Standard Soap Solution.—Castile soap, which is supposed to be made with soda and olive oil, is much used for standard soap solutions, but the writer has found that it is liable to considerable deterioration on keeping, especially in cold weather, owing to the deposition of stearate and palmitate of soda; and warming up the solution with a view to their re-solution makes it more prone to deposit a fresh crop of precipitate, and in consequence it is never safe to use the solution unless "*standardized*" previous to use, which necessitates undesirable factors. Sodid oleate, supplied by Hopkin and Williams, London, makes a standard soap solution which suffers very little change on keeping, and can be generally recommended for the purpose. About 13 grms. of it are dissolved in a mixture of 500 cc. of methylated alcohol and 500 cc. of water, and filtered if necessary. It now becomes necessary to standardize it, so that 1 cc. will be equivalent to .001 gram. of carbonate of lime. In order to effect this, 12 cc. of the standard hard water are run into a 250 cc. stoppered bottle from a burette and diluted to 70 cc. A burette is now filled with the soap solution, which is run into the bottle 1 cc.

at a time, and the bottle vigorously shaken after each addition, until a point is reached where a persistent lather, lasting for at least five minutes, is obtained. Note the volume required. 12 cc. of hard water should require 13 cc. of soap solution (distilled water itself requiring 1 cc. to form a lather), but it will be a figure less than this, and therefore the soap solution is too strong, and will require diluting, so that 12 cc. of standard "hard" water will require 13 cc. of soap solution. An example of an actual preparation of a standard soap solution will make matters clear:—

13 grms. of sodic oleate were dissolved in a mixture of 500 cc. of methylated alcohol and 500 cc. of water, and filtered. On testing in the manner described, 12 cc. of the standard "hard" water, diluted to 70 cc., required 11.4 cc. of the soap solution to form a persistent lather.

Now, since 13 cc. should have been required, every 11.4 cc. of the soap solution left, requires diluting by $13 - 11.4 = 1.6$ cc.

There were 960 cc. of the solution left, therefore $\frac{960}{11.4} = 84.2$, and $84.2 \times 1.6 = 134.7$ cc. more of the mixture of alcohol and water to be added.

On adding this quantity, thoroughly mixing, and testing as before, 12 cc. of the standard hard water required exactly 13 cc. of the soap solution.

Estimation of Total Hardness.—70 cc. of the clear sample are run into a 250 cc. stoppered bottle, and the standard soap solution run into it in the manner described above, until a lather capable of persisting for five minutes is produced. The number of cubic centimetres required, minus 1 cc. for the water itself, will give the hardness in degrees Clark, or in terms of chalk in grains per gallon. If the water should require more than 16 cc. of standard soap solution, 70 cc. of distilled water must be added, and the testing continued as before, in order that the lathering may take place uniformly. Or 35 cc. of the sample are taken, diluted to 70 cc., and tested as before; but the result must be multiplied by 2. If the water contains a fair proportion of magnesia salts, there will be a little difficulty in

hitting the right point, owing to the slowness with which magnesia salts decompose soap; an apparent persistent lather is formed, which on being allowed to stand a little while and again shaken up, will disappear; a little experience with magnesian hard waters will familiarize the operator with this peculiarity.

The Permanent Hardness.—250 cc. of the clear sample are poured into a 500 cc. flask, and boiled for one hour, the original volume being kept up by frequent additions of boiling distilled water free from CO_2 ; after which it is covered over with a watch glass and put into cold water for a quarter of an hour to cool. It is now quickly poured into a 250 cc. graduated stoppered flask, diluted if necessary to exactly 250 cc. at 15°C. with distilled water free from CO_2 , well mixed, and filtered. 70 cc. of the well-mixed solution are now poured into the bottle, and the permanent hardness determined as described.

The Temporary Hardness.—The temporary hardness, or that hardness removed by boiling, is obtained by deducting the degree of permanent hardness from that of the total.

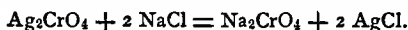
Estimation of Chlorine in Chlorides.—Chlorine is mostly present in water as common salt (NaCl), but it is sometimes present as potassic chloride (KCl), calcium chloride (CaCl_2), and magnesium chloride (MgCl_2), so that it is wise to express the chlorine as chlorides, which of course could be run out in terms of common salt. The chlorine in waters is usually determined volumetrically by a standard solution of argentic nitrate, using potassic chromate as indicator of the end of the reaction.

Preparation of Standard Silver Nitrate Solution.—Dissolve 4.79 grms. of pure nitrate of silver in a little distilled water, dilute to exactly 1,000 cc. at 15°C. and thoroughly mix. Each cc. of this solution will exactly precipitate .001 gm. of chloride as chloride of silver.



Method.—Measure out 70 cc. of the sample into a porcelain

basin of about 100 cc. capacity, and add to it two drops of E potassic chromate solution. Fill a burette with the standard silver nitrate solution, and run this gradually into the sample, stirring until the last drop added turns the solution from yellow to yellowish-red, owing to the formation of silver chromate. The principle of the process depends upon the fact, that silver has a greater affinity for chlorine than for chromic acid, and chromate of silver in consequence is not permanently produced until all the chlorine is precipitated, although when the silver solution drops into the sample tinted with potassic chromate, the silver chromate is momentarily formed, but is decomposed on stirring the solution, by the chlorides in excess, thus :



The number of cubic centimetres required for 70 cc. minus 0.1 cc. taken up for colouring this quantity of distilled water will represent the chlorine in terms of grains per gallon, and this multiplied by $\frac{117}{71}$ will give the number of grains in terms of common salt (NaCl) per gallon.

Determination of Alkalinity.—By alkalinity is understood the power of neutralizing an acid, and by noting the amount of standard acid of known strength required to neutralize a definite volume of water, the degree of alkalinity can be ascertained.

Preparation of Standard $\frac{E}{10}$ H_2SO_4 .—Run out from a burette 50 cc. of standard E H_2SO_4 into a graduated 500 cc. flask, dilute with distilled water to 500 cc. at 15° C., and thoroughly mix. 1 cc. of this solution will exactly neutralize .005 grm. of carbonate of lime or .0053 grm. of carbonate of soda.

Preparation of Cochineal Solution.—Grind about 5 grms. of cochineal in a mortar, brush into a 300 cc. beaker, pour on 200 cc of water and 50 cc. of methylated alcohol, and allow to

digest with frequent stirring for a few hours at about 60° C. Filter and bottle.

Method.—Measure off 250 cc. of the sample into a 300 cc. flask, add 2 cc. of cochineal solution, and mix; if carbonates be present in the water a pink colour will be produced. Treat 250 cc. of distilled water in the same manner, when a yellow solution is produced, which will be of service in ascertaining by comparison the colour indicating the end of the reaction with the $\frac{E}{10}$ H_2SO_4 on the sample. Fill a burette with the standard $\frac{E}{10}$ H_2SO_4 , run it into the sample gradually, stirring, until the last drop turns the solution from pink to the colour of the tinted distilled water, and note the volume of solution required. It is customary to express the alkalinity in terms of grains of carbonate of lime per gallon, and the number of cubic centimetres of $\frac{E}{10}$ H_2SO_4 required for 250 cc. of water, multiplied by .005 and 280, will give the desired figure.

Method for the Complete Mineral Analysis of a Water.—*Estimation of Silica, Oxide of Iron and Alumina, Lime and Magnesia.*—Acidulate 1,000 cc. of the filtered sample with 10 E HCl, evaporate to complete dryness in a platinum or porcelain dish, and heat the residue to about 130° C. for about an hour, to render silica insoluble. Allow the dish to cool, moisten residue with 5 cc. of 5 E HCl, add 50 cc. of water, and allow to digest until all that is soluble is dissolved; filter off the insoluble residue, which is silica, through a small ashless filter, wash with hot water till free from chlorides, dry, ignite in weighed crucible, cool, and weigh; increase in weight multiplied by 70 = grains per gallon of silica. The solution is now made slightly alkaline with 20 E AmHO, gently boiled until the smell of ammonia has nearly all gone off, the precipitated oxide of iron and alumina filtered through a small ashless filter, washed, dried, ignited and weighed. The proportion of oxide of iron and alumina in water is usually

so small that they are estimated and expressed together, and sometimes with the silica in addition. The filtrate from the iron oxide, &c., which should be about 150 cc. in volume, is now made strongly alkaline with, say, 5 cc. of 20 E AmHO, and 5 cc. of 5 E AmCl are added to keep magnesia in solution. 10 cc. of $\frac{E}{2}$ ammonic oxalate are now added, the solution well stirred, and the precipitated oxalate of lime thus produced allowed to subside for a few hours, after which it is filtered off through an ashless filter, washed with water, dried in the water bath, transferred to a weighed platinum crucible, heated gently at first, and finally for ten minutes to the highest heat of the blow-lamp. The oxalate thus becomes converted into anhydrous calcium oxide (lime); cool in the desiccator, and weigh. Increase in weight multiplied by 70 = grains of combined lime per gallon, and this figure multiplied by 0.7143, gives grains of calcium per gallon.

The filtrate is now evaporated to about 80 cc., 5 cc. of 20 E AmHO and 6 cc. of E sodic phosphate solution added, well stirred, and allowed to stand in the cold for about twelve hours. The magnesia is thus precipitated as ammonio-phosphate. It is filtered off, and the filtrate used with the aid of a feather to transfer the last portions of precipitate sticking to the beaker, to the filter. This method is adopted to limit the amount of wash water used, in which the precipitate is somewhat soluble; the precipitate is finally washed with a minimum quantity of 10 E AmHO. The filtrate is measured, and .001 gm. allowed for each 50 cc., due to its solvent action on the ammonio-phosphate. The precipitate is dried, carefully ignited to a bright red heat in a weighed platinum or porcelain crucible, cooled, and the resulting pyrophosphate of magnesia ($Mg_2P_2O_7$) weighed. After adding the amount that was dissolved by the ammoniacal solution, multiply by .2162 and 70, and the figure obtained expresses grains per gallon of magnesium.

Estimation of Potash and Soda.—1,000 cc. of the sample are evaporated to about 50 cc., about 0.15 gm. of caustic baryta added, the solution boiled for a little while, and the precipi-

tated carbonates, hydrate of magnesia, &c., filtered off. 5 E Am_2CO_3 is now gradually added until no further precipitate of carbonates of baryta and lime is produced. After allowing to settle, they are separated by filtration, the filtrate being received in a weighed platinum dish.

The solution now contains the sodium and potassium in the form of chlorides, together with ammoniacal salts; it is acidulated with 10 E HCl, evaporated to dryness, and the residue cautiously heated with the naked flame of a Bunsen burner to expel ammoniacal salts, which are driven off as dense fumes. Great care must be taken that the heat applied does not exceed very dull redness, or the chlorides of the alkalies will suffer loss by volatilization. After fumes have ceased to come off, the dish containing now KCl + NaCl is cooled in a desiccator and weighed. If the proportion of K and Na present be required, proceed as follows:—Dissolve in 10 cc. of water, add 4 cc. of E. platinic chloride, evaporate on water bath to a pasty condition, digest with 10 cc. of methylated alcohol, decant off the clear liquid from the precipitate of double chloride of platinum and potassium (2KCl.PtCl_4) through a weighed filter, add 5 cc. more alcohol to the residue, digest, and decant as before, repeating the digestion a third and fourth time, finally transferring the precipitate to the filter with the aid of a feather and a stream of alcohol from a small wash bottle. The filter and its contents are now dried in a water oven and weighed between two tared watch glasses. The weight of the 2KClPtCl_4 thus obtained, multiplied by $0.3070 = \text{KCl}$; subtract this from the weight of KCl + NaCl as obtained above; remainder = NaCl. Then—

$\text{KCl} \times .5244 \times 70 = \text{grains K per gallon,}$
and

$\text{NaCl} \times .3934 \times 70 = \text{,, Na ,, ,,}$

Estimation of Sulphates—1,000 cc. of the clear sample are acidulated with 10 cc. of 10 E HCl, evaporated to about 250 cc. in a porcelain basin, transferred to a 300 cc. beaker, heated to boiling, 10 cc. of E baric chloride solution added, well stirred, and allowed to stand twelve hours. The resulting

baric sulphate is then filtered off, washed, dried, scraped in a weighed crucible, filtered, ignited separately, residue added to main portion, ignited at a dull red heat for ten minutes, cooled and weighed. $\text{BaSO}_4 \times .412 \times 70 =$ grains combined SO_4 (sulphur) per gallon.

Estimation of Nitrates.—A very neat method of estimating the proportion of nitrates and nitrites in waters, is the adoption of Crum's method for the refraction of nitre, in which process the determination is deduced from the volume of nitric oxide

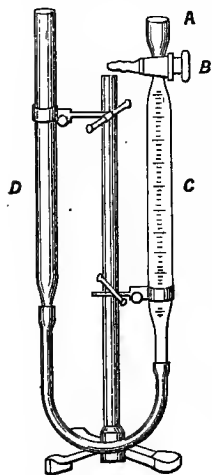


FIG. 20.

gas given off when a concentrated solution of the sample is shaken up with 36 E H_2SO_4 and mercury, in a graduated tube termed a nitrometer. The following is the manner in which the process is carried out:—500 cc., or more if necessary, of the sample, are evaporated to dryness in a porcelain basin, and the residue drenched repeatedly with small quantities of hot water, the solution of the nitrates being assisted by rubbing the residue with the clean finger. Filter into a small beaker, and wash the residue with a little hot water to ensure complete solution of all the soluble nitrate. The solution is now to be evaporated to a volume not exceeding $1\frac{1}{2}$ cc., and poured into the cup, A, of the nitrometer, Fig. 20, already filled with mer-

cury. By lowering the tube D, and opening the tap B, the liquid is made to run into the graduated tube C, and by careful manipulation no air need enter; wash out the beaker with small quantities of water into C (using 3 cc. in all), and then pour 6 cc. of 36 E H_2SO_4 free from nitrates into the beaker, finally transferring it in small quantities at a time into C through B. Any air or CO_2 which is produced is now got rid of by rising D and carefully opening B. The tube, C, is now taken out of its clip, held at an angle of about 40° , and shaken in this plane.

for about five minutes, part of the mercury being thus finely divided and mixed with the liquid. Allow to stand for five minutes, and again shake as before, until no further increase in the volume of gas given off takes place. Raise or lower D, as the case may be, until the level of the mercury therein is about one-seventh of the volume of the liquid above the level of the mercury in C. Read off the volume of nitric oxide (NO), and note the temperature and barometric pressure at the time.

Now since nitric oxide contains half its volume of nitrogen, the volume of gas thus found represents the volume of nitrogen from 1000 cc. of sample; 1000 cc. of nitrogen at 0° and 760 mm. mercury weigh 1.2544 grms., so that

$$\frac{\text{cc. of N (at 0° C. and 760 mm.)} \times 1.2544 \times 70}{1,000} =$$

grains of nitrogen per gallon, and the figure thus obtained multiplied by $\frac{62}{14}$ = grains NO_3 per gallon.

Estimation of Organic Matter.—The accurate determination of total organic matter in a water is attended with difficulties, owing to the decomposition of carbonate, nitrate, magnesian chloride, &c., during the ignition of the residue. However, a fair idea may be had of the proportion present by the following method:—Evaporate 500 cc. of the water to dryness in a platinum dish, and dry the residue at 130° C. until the weight remains constant; we thus obtain the total solid residue. It next becomes necessary to gently ignite so as to burn off all organic matter at as low a temperature as possible. For this purpose the dish is placed on a pipeclay triangle, fixed to a ring on a retort stand, and a flame, from a Bunsen burner, held in the hand, is made to play on the dish for ten minutes, care being taken that the temperature does not exceed a dull red heat. It is now cooled in a desiccator and weighed, and the ignition repeated as before until no further loss occurs. The CO_2 that has been driven off from the carbonates is now replaced by evaporating with strong CO_2 water, prepared by acting upon marble with 2 F. HCl, washing the gas with water,

and saturating distilled water with the washed gas. The dish is filled with CO_2 water thus prepared, and evaporated to dryness, the process being repeated four times more, which will be generally sufficient to replace all CO_2 lost by ignition. The residue is next dried at 130°C . until the weight remains constant. By subtracting this weight from that of the total solid residue the remainder is taken as organic matter.

Conversion of Solid Residue into Sulphates.—Often it is sufficiently accurate to determine the amount of combined sodium (disregarding potassium) indirectly. For this purpose the residue from the determination of the organic matter is evaporated to dryness with excess of E sulphuric acid to convert into sulphates, the residue gently ignited with ammoniac carbonate, cooled, and weighed. The following analytical data of a complete analysis of a sample of water from the river Wye, at Ross, Monmouthshire, by the writer, illustrate the manner in which the bases and acids are combined, the combined sodium being in this case determined indirectly.

RIVER WYE WATER.

Estimation of Silica, Oxide of Iron, and Alumina.

1,000 cc. taken.

Porcelain Crucible marked 4	8.2225
Do. + SiO_2 + Fe_2O_3 + Al_2O_3	8.2262
		<hr/>
		.0037

$$.0037 \times 70 = 0.259 \text{ grain per gallon.}$$

Estimation of Combined Calcium.

1,000 cc. taken.

Porcelain Crucible, marked 1	6.4129
Do. + CaO + filter ash No. 3	6.4450
„ „ „ 2nd ignition	6.4449
		<hr/>
CaO + F.A.0320
F.A.0017
		<hr/>
CaO0303

$$.0303 \times .7143 \times 70 = 1.52 \text{ grains CaO per gallon.}$$

Estimation of Combined Magnesium.

1,000 cc. taken.

Porcelain Crucible, marked 2	6.1902
Do. + $Mg_2P_2O_7$	6.2052
„ „ 2nd ignition	6.2052
		<hr/>
		.0150
Allowing for solubility of precipitate in ammoniacal solution0026
		<hr/>
	$Mg_2P_2O_7$. .	.0176

$$.0176 \times .2162 \times 70 = 0.27 \text{ grain Mg. per gallon.}$$

Estimation of Sulphates (SO_4).

1,000 cc. taken.

Porcelain Crucible, marked B	6.4129
Do. + $BaSO_4$ + F.A. No. 2	6.4412
„ „ „ „ 2nd ignition	6.4412
		<hr/>
		.0283
	F.A. . .	.0009
		<hr/>
		.0274

$$.0274 \times .412 \times 70 = 0.79 \text{ grain } SO_4 \text{ per gallon.}$$

Estimation of Chlorine in Chlorides.

100 cc. taken.

1 cc. standard $AgNO_3 = .001$ gram. Cl.100 cc. sample required .95 cc. standard $AgNO_3$. $.95 \times .7 = .67$ grain chlorine per gallon.

$$\text{or } \frac{.67 \times 117}{71} = 1.1 \text{ grain common salt (NaCl) per gallon.}$$

Total Hardness.

70 cc. of the sample required 6 cc. of standard soap solution to produce a persistent lather for 5 mins. $6 - 1 = 5^\circ$ Clark.

Estimation of Nitrates.

500 cc. taken.

NO liberated	0.2 cc.
Temperature	19° C.
Barometric pressure	29.25"

Tabulated Results.

	Grains per gall.
Organic matter	0.27
Inorganic matter	6.90
Total Sulphate formed	8.06
<hr/>	
Calcium	1.52
Magnesium	0.27
Silica, Oxide of Iron, and Alumina	0.26
Sulphion (SO ₄)	0.79
Chlorine	0.67
Sodium (by calculation)	0.41
Nitron (NO ₃)	0.07
Carbonates (CO ₃)	2.41
	<hr/>
	6.40

and these may be combined as follows :—

$$\frac{1.52 + 17}{5} = 5.17 \text{ CaSO}_4.$$

$$.27 \times 5 = 1.35 \text{ MgSO}_4.$$

$$\text{CaSO}_4 + \text{MgSO}_4 + \text{SiO}_2, \text{ \&c.} = 6.78.$$

$$8.06 - 6.78 = 1.28 \text{ Na}_2\text{SO}_4.$$

$$71 : 23 :: 1.28 : x = .41 \text{ Na.}$$

$$46 : 117 :: .41 : x = 1.04 \text{ NaCl.}$$

$$1.04 - .41 = .63 \text{ combined Cl used.}$$

$$.67 - .63 = .04 \text{ Cl remaining.}$$

$$71 : 111 :: .04 : x = .06 \text{ CaCl}_2.$$

$$.06 - .04 = .02 \text{ Ca used.}$$

$$1.52 - .02 = 1.5 \text{ Ca remaining.}$$

$$31 : 41 :: .07 : x = .09 \text{ Ca (NO}_3)_2.$$

$$.09 - .07 = .02 \text{ Ca used.}$$

$$1.5 - .02 = 1.48 \text{ Ca remaining.}$$

$$12 : 17 :: .79 : x = 1.12 \text{ CaSO}_4.$$

$$1.12 - .79 = .33 \text{ Ca used.}$$

$$1.48 - .33 = 1.15 \text{ Ca remaining.}$$

$$2 : 5 :: 1.15 : x = 2.88 \text{ CaCO}_3.$$

$$2 : 7 :: 0.27 : x = .95 \text{ MgCO}_3.$$

Summary.

	Grains per gall.
Sodic Chloride	1·04
Calcic Chloride	0·06
Calcic Nitrate	0·09
Calcic Sulphate	1·12
Calcic Carbonate	2·88
Magnesium Carbonate	0·95
Silica, Oxide of Iron, and Alumina	0·26
Organic Matter	0·27
	<hr/>
	6·67

Remarks.—Solids gave a slight odour of peat on ignition. Colour (before and after filtration) brownish-yellow. Suspended matter very small. Reaction : very slightly alkaline.

Owing to the small proportion of incrustating salts present, this water is of first class quality for use in steam boilers.

The following ingredients were obtained by Stillman in a sample of water by the method described on the opposite page :—

	Grains per U.S. gallon.
Silica	0·4771
SO ₃	1·2012
Cl	0·3206
K ₂ O	0·0291
Na ₂ O	0·3615
MgO	0·4490
CaO	1·1313
Fe ₂ O ₃ Al ₂ O ₃	0·2973
Organic	1·1254
Carbonic Acid	0·7989
	<hr/>
	6·1914
Oxygen in excess of Cl	0·0932
	<hr/>
	6·0982

Having determined the component parts of the water residue in grains per gallon, it becomes necessary to unite these in chemical union, as near as possible, as they exist in the water.

The general rule may be stated as follows :—The chlorine is combined with the sodium ; if still in excess, with the potassium, then magnesium, and finally calcium. The sulphuric acid is combined to the alkalies, provided there is not enough

chlorine to saturate them; then to the calcium, and finally to the magnesium.

The carbonic acid is united with the calcium and magnesium after the other combinations are made. There are exceptions to this rule; mineral waters and many artesian well waters forming notable examples.

Carrying out the above, the following is obtained:—

	Grms. per litre.	Grains per U.S. gallon.
NaCl	0·0091	0·5306
Na ₂ SO ₄	0·0033	0·1923
K ₂ SO ₄	0·0009	0·0524
CaSO ₄	0·0311	1·8136
CaCO ₃	0·0118	0·6880
MgCO ₃	0·0162	0·9446
Fe ₂ O ₃ Al ₂ O ₃	0·0051	0·2973
SiO ₂	0·0082	0·4771
Organic, &c.	0·0193	1·1254
Total	0·1050	6·1213

This analysis shows, that the principal scale-forming ingredient is calcium sulphate, being more than equal to the carbonates of calcium and magnesium.

The following analysis is of a water containing sulphuric acid, but the alkalis being present in sufficient amount to combine with all of it, as well as the chlorine, no sulphate of lime is present.

	Grms. per litre.	Grains per U.S. gallon.
SiO ₂	0·0038	0·2215
SO ₃	0·0110	0·6414
Cl	0·0062	0·3615
K ₂ O	0·0033	0·1923
Na ₂ O	0·0185	1·0788
MgO	0·0165	0·9388
CaO	0·0466	2·7175
Al ₂ O ₃ Fe ₂ O ₃	0·0020	0·1166
Organic Matter	0·0246	1·4345
Carbonic acid	0·0530	3·0908
	0·1855	10·7937
Oxygen in excess of Cl	0·0021	0·1224
Total	0·1834	10·6713

Combined as follows :—

	Grms. per litre.	Grains per U.S. gallon.
NaCl	0·0154	0·8900
Na ₂ SO ₄	0·0141	0·8223
K ₂ SO ₄	0·0061	0·5557
CaCO ₃	0·0833	4·8577
MgCO ₃	0·0338	1·9710
Al ₂ O ₃ Fe ₂ O ₃	0·0020	0·1166
SiO ₂	0·0038	0·2215
Organic	0·0246	1·4345
Total	0·1831	10·8693

Where all the chlorine is not in combination with the sodium and potassium, chloride of magnesium is usually present.

This latter compound, while not scale-forming, is considered an active corrosive agent—upon the supposition that at the temperature of 100° C., and higher, it is decomposed, and hydrochloric acid formed and liberated.

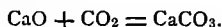
The report, given below, is of a water from a driven well in Florida. Complaint having been made that not only was the scale excessive in amount, but that corrosive action was also very marked, an analysis was made, reference to which readily explains the difficulty encountered in the boilers.

	Grms. per litre.	Grains per U.S. gallon.
NaCl	0·323	18·87
KCl	0·067	3·91
MgCl ₂	0·104	6·06
CaSO ₄	0·197	11·52
CaCO ₃	0·293	17·10
MgCO ₃	0·144	8·40
SiO ₂	0·011	0·62
Al ₂ O ₃ Fe ₂ O ₃	0·007	0·46
Organic	0·138	8·02
Total	1·284	74·86

In all of the above analyses the constituents have been stated in grains per U.S. gallon of 231 cubic inches, rather than in parts per 100,000, the former being in general use by the me-

chanical profession as the proper method by which to express the weights of the component parts of water.

The Softening of Hard Waters.—Waters that are excessively hard, and due to “temporary hardness,” that is, hardness due to the carbonates of lime and magnesia held in solution by excess of carbonic acid, and which can be removed by the boiling of the water, may be conveniently softened by Dr. Clark’s process, the principle of which depends upon the formation of carbonate of lime, brought about when hydrate of lime (lime water), is added to such a water. The lime in the lime water combines with the free or incipiently combined carbonic acid gas, thus :—



when all except a little over 2 grains per gallon of the carbonate of lime and magnesia are precipitated, which can be separated by filtration or decantation. In calculating the amount of quicklime—which is to be “slaked”—necessary to remove the temporary hardness from a water, Dr. Frankland states that, “To soften 700 gallons of water, 1 oz. of quicklime is required for each part of temporary hardness in 100,000 parts of water.”

The following is an example of the manner in which the amount of lime required is calculated, together with its cost, to remove the temporary hardness from 1,000 gallons of water :—

A sample of water had 14·7 degrees of temporary hardness.

70,000 : 100,000 :: 14·7 : x = 21 parts temporary hardness per 100,000 parts.

21 oz. lime is therefore required for 700 gallons and
700 : 1,000 :: 21 : x = 30 oz. for 1,000 gallons.

$\frac{30}{16} = 1\cdot88$ lbs. Best lime costs 12/6 per ton of 2,240 lbs.

12/6 = 150 pence.

2,240 : 1·88 :: 150 : x = 0·13 pence per 1,000 gallons of water.

The permanent hardness, due principally to sulphates of lime

and magnesia, can nearly all be removed by adding sodic carbonate to the water after the lime treatment, calcium carbonate and soluble sodic sulphate being formed thus:—



but the cost of this process is considerably more than that of the removal of temporary hardness by the lime treatment, and if the CaSO_4 be present in large quantities, its equivalent of sulphate of soda formed would cause "priming" in boilers, which would be very objectionable.

Analyses of River Waters.—The following are analyses of various river waters, expressed in parts per 100,000. :—

THE THAMES AT TWICKENHAM.

Total solid contents 32·01.

Carbonate of Lime . . .	18·23	Sulphate of Potash . . .	0·95
„ Magnesia . . .	1·47	Chloride of Lime . . .	2·50
Sulphate of Lime . . .	0·64	Silica	0·39
„ Soda	2·86	Organic Matter	4·97

THE SEINE ABOVE PARIS.

Total solid contents 17·90.

Carbonate of Lime . . .	9·20	Sulphates of Soda and Mag-	
„ Magnesia . . .	3·90	nesia	1·00
Sulphate of Lime . . .	2·00	Chloride of Lime . . .	1·00
		Silica, Alumina, Iron Oxide	0·80

THE RHINE AT STRASSBURG.

Total Solid contents, 23·18.

Carbonate of Lime . . .	13·56	Nitrate of Potash . . .	0·38
„ Magnesia . . .	0·51	Silica	4·88
Sulphate of Lime . . .	1·47	Alumina	0·25
„ Soda	1·35	Iron Oxide	0·58
Chloride of Soda . . .	0·20		

THE RHONE AT GENEVA.

Total solid contents 18·20.

Carbonate of Lime . . .	7·89	Chloride of Soda . . .	0·17
„ Magnesia . . .	0·49	Nitrate of Soda . . .	0·85
Sulphate of Lime . . .	4·66	Silica	2·38
„ Magnesia . . .	0·63	Alumina	0·39
Sulphate of Soda . . .	0·74		

THE DANUBE AT VIENNA.

Total solid contents 12·62.

Carbonate of Lime . . .	8·37	Sulphate of Potash and Soda	0·20
„ Magnesia . . .	1·50	Silica	0·49
Sulphate of Lime . . .	0·29	Iron Oxide	0·20
Sulphate of Magnesia . . .	1·57		

THE SPREE AT BERLIN.

Total solid contents 11·40.

Carbonate of Lime . . .	6·50	Chloride of Soda . . .	1·20
„ Magnesia . . .	0·90	Nitrate of Soda . . .	0·30
Sulphate of Soda . . .	0·60	Alumina and Iron . . .	1·30
Sulphate of Potash . . .	0·60		

The following are analyses by Professor Wanklyn, of waters supplied to the Metropolis by three of the principal water companies :—

WEST MIDDLESEX COMPANY.

(Grains per gallon).

Silica	0·3	Sulphate of Magnesia . . .	0·6
Carbonate of Lime . . .	12·9	Nitrate of Magnesia . . .	1·1
Sulphate of Lime . . .	2·4	Chloride of Sodium . . .	2·0

NEW RIVER COMPANY.

Silica	0·26	Nitrate of Lime . . .	1·00
Alumina, &c.	0·14	„ Magnesia	1·28
Carbonate of Lime . . .	12·70	Chloride of Sodium . . .	2·02
Sulphate of Lime . . .	1·60		

KENT COMPANY.

Silica	0.75	} = 18.3 insoluble solids.
Alumina, &c.	0.22	
Carbonate of Lime	16.30	
Water	1.03	
Silica, Alumina, &c.	0.28	} = 12.0 soluble solids.
Sulphate of Lime	5.37	
„ Magnesia	0.93	
Nitrate of Magnesia	1.20	
„ Soda	1.21	
Chloride of Soda	2.64	
Water	0.37	

DEEP WELL WATER AT CROYDON, SURREY.

(Grains per gallon).

Total solid contents	21.6	Sulphate of magnesia	1.4
Silica	trace	Chloride of soda	1.8
Carbonate of lime	14.1	Nitrate of soda	1.4
Sulphate of lime	1.8	„ potash	1.1

Sea Waters.—The following are analyses of various sea waters, from which it is seen that sodic chloride is present to the extent of nearly 3 per cent., the other constituents being comparatively small.

	British Channel. (Schweitzer). Pts. per 1000.	Irish Sea. (Thorpe). Pts. per 1000.	Mediterranean. (Usiglio). Pts. per 1000.
Water	963.74372	966.14054	962.345
Sodic Chloride	28.05948	26.43918	29.424
Potassic Chloride	0.76552	0.74619	0.505
Magnesian Chloride	3.66658	3.15083	3.219
Magnesian Bromide	0.02929	0.07052	0.556
Magnesian Sulphate	2.29578	2.06608	2.477
Calcic Sulphate	1.40662	1.33158	1.357
Calcic Carbonate	0.03301	0.04754	0.114
Iodine	traces	—	—
Ammonic Chloride	„	0.00044	—
Ferrous Carbonate	—	0.00503	0.003
Magnesian Nitrate	—	0.00207	—
	1000.00000	1000.00000	1000.000
Specific Gravity	1027.4 at 16°	1024.84 at 15°	1025.8 at 21°

According to Dittmar the solids obtained from sea water have the following percentage composition :—

Chloride of Sodium	77.76
Chloride of Magnesium	10.88
Sulphate of Magnesia	4.74
Sulphate of Lime	3.60
Sulphate of Potash	2.46
Bromide of Magnesium	0.22
Carbonate of Lime	0.34
	<hr/>
	100.00

II. WATER FOR DRINKING PURPOSES. .

No doubt many of the diseases that human flesh is heir to, are propagated through the drinking of water contaminated with those microscopic creatures which, from time to time, have been proved to be the *causa causans* of such maladies. It is essential, therefore, that the water we drink should be free from those sources of danger. Water should likewise be as free as possible from unorganized organic matter, and a limited amount only of mineral constituents should be present.

In judging of the suitability of a water for general domestic use, the following data will usually be found sufficient to form an opinion as to its quality:—Total Solids; Chlorine; Free and Albuminoid Ammonia; Nitrites and Nitrates; Hardness; Poisonous Metals; Colour before and after Filtration; Suspended matter; and The Odour of the Water when warmed. In some cases, such as the question of a town supply, a complete mineral analysis will be required in addition.

The Total Solids.—These may be determined as directed on page 148. They should be gently ignited; and an observation of the odour given off will serve to show whether any organic matter present is of animal or vegetable origin. With

regard to the amount of total solid matter present, which may allow the water to be pronounced fit for drinking, Dr. Wanklyn states that if it does not exceed 30 or 40 grains per gallon there would be no reason for rejecting it on that score.

The Chlorine.—The chlorine is to be determined as directed on page 151. The amount of chlorine present in a sample of water proposed to be used for drinking forms an important datum, since it is, within certain limits, an indication of sewage contamination, animal secretions being highly charged with salts in which chlorine is a heavy constituent, whereas natural waters usually contain but very small proportions of chlorine, often only one grain in the gallon. Although a water may contain a comparatively high proportion of chlorine, it does not *always* follow that it is charged with sewage since the geological strata through which the water passes may afford it an unusually high amount of chlorine. A water containing little or no chlorine is sure not to contain any sewage, but at the same time it may be charged with objectionable decomposing vegetable organic matter, so that the amount of chlorine present, as seemingly indicating presence or absence of organic matter, must be taken with some reservation. The amount of chlorine in sewage is from 9 to 12 grains per gallon.

Free and Albuminoid Ammonia.—The estimation of these bodies, known as Wanklyn's ammonia process, affords valuable criteria as to the purity of a water, organically considered. The "free" ammonia is that which can be removed by simple distillation of the water, while the "albuminoid" ammonia does not pre-exist as ammonia in waters, but is produced by the conversion of the nitrogen, contained in albuminous or nitrogenized organic matter present, by the action of a solution of potassium permanganate, made strongly alkaline with potassic hydrate. The whole of the nitrogen is not liberated from albuminoid substances as ammonia by this means, but the amount given off is a fraction of the total, being thus pro-

portional to the quantity of nitrogenous or albuminoid substances present.

The ammonia process requires the following standard solutions, &c. :—

Dilute Standard Solution of Ammonia.—Two solutions are generally kept: a strong solution which contains 1 milligram of ammonia in each cubic centimetre, and a weak solution prepared by diluting 1 volume of the former to 100 volumes, which will thus contain $\frac{1}{100}$ of a milligram of ammonia in each cubic centimetre. The strong solution is prepared by dissolving 3.15 grms. of pure dry ammonic chloride in water, diluting to 1,000 cc. at 15° C., and well mixing; and to prepare the weak solution 10 cc. of the solution thus prepared are run from a burette into a graduated litre flask, and diluted to 1,000 cc. at 15° C.

Alkaline Potassic Permanganate Solution.—Weigh out 8 grms. of potassic permanganate and 200 grms. of stick potassic hydrate, into a $1\frac{1}{2}$ litre basin, add 1,000 cc. of distilled water free from ammonia, and stir until all is gone into solution; the solution is now boiled, until about one quarter of the liquid has boiled off, diluted to original bulk, again boiled down as before, and finally diluted to 1,000 cc. and bottled. It is necessary to give the solution a thorough boiling in order to drive off any ammonia that may be present. Mere evaporation below the boiling point does not appear to be very effective in removing any traces of NH_3 present. 50 cc. of this solution are required for each analysis, and the amount of ammonia still left in this quantity must be determined by the method herein described, using water free from ammonia in the distillation. If any appreciable quantity of ammonia be thus found, it must be deducted from the amount obtained for albuminoid ammonia in each analysis.

The Nessler Reagent.—This extremely delicate test for ammonia is prepared as follows:—Weigh out 35 grms. of potassic iodide and 13 grms. of mercuric chloride (*corrosive sublimate*) in a $1\frac{1}{2}$ litre beaker, dissolve in about 800 cc. of hot distilled water, and allow to cool; a saturated solution of mercuric chloride is now cautiously added until a slight but

permanent precipitate of red periodide of mercury is produced, the solution being, of course, well stirred during the addition. 160 grms. of potassic hydrate or 120 grms. of sodic hydrate are now added to the liquid and stirred until it all goes into solution, which is finally to be diluted to 1,000 cc. The solution is now to be rendered sensitive by adding at first 2 cc. of saturated mercuric chloride solution, well shaken, and the precipitate allowed to settle. On adding 2 cc. of Nessler reagent to 50 cc. of water containing 0.5 cc. of the dilute ammonia solution, an immediate yellowish-brown colour should be produced, owing to the formation of dimercur-ammonium iodide $\text{NH}_2\text{I}_2\text{OH}_2$. If this is not the case the solution is not sufficiently sensitive, and more mercuric chloride solution must be added until the desired delicacy is produced.

Distilled Water free from Ammonia.—It is very obvious that, in testing for such minute quantities of ammonia, the water used for diluting, &c., should be free from all traces of ammonia, or must not contain more than .005 milligram in 100 cc. The writer prepares distilled water free from ammonia, by distilling good river water. The first 10 per cent. of distillate is rejected; the next 30 per cent. put into the ordinary stock bottle, the next 20 per cent. is bottled separately, and will be found tolerably free from ammonia. The rest of the distillate, which contains traces of ammonia, is put with the ordinary stock.

It is needless to remark that all apparatus must be scrupulously clean for this process, and that all bottles or any other vessel containing solutions from which ammonia is liable to be given off must be kept out of the room for the time the analysis is on, or locked in a cupboard some distance away, or placed in a good stink closet, provided with a good draught.

Estimation of Free Ammonia.—A stoppered litre glass retort is selected, and about 10 cc. of strong sulphuric acid poured into it, which is allowed to moisten the whole of the interior of the retort, after which it is washed out with plenty of tap water until the washings no longer redden blue litmus paper. It is next fixed to a Liebig's

condenser by means of a piece of stout india-rubber tubing, that has been previously boiled for some time in a $\frac{E}{2}$ solution of sodic hydrate, in order to free it from any traces of ammonia. About half a dozen pieces of recently-ignited pumice stone each about the size of a pea are now introduced into the retort, which serve to prevent "bumping," and 500 cc. of water free from ammonia poured in. Any water present in the condenser is allowed to run off, so as to allow *steam* to pass through to clear out all traces of ammonia. A Bunsen burner is lit under the retort and the water made to boil; steam will soon find its way through the condenser, and after it has been steaming for about half an hour, the cold water is turned on to the condenser and a Nessler cylinder (these are colourless glass cylinders usually 6 ins. high and $1\frac{1}{2}$ ins. in diameter, and marked at 50 cc.) is now put under the delivery tube. When 50 cc. have come over, add to it 2 cc. of the Nessler solution and mix by pouring backwards and forwards into an empty Nessler glass. On placing on a sheet of white paper there should be no yellow coloration produced; if there should be, continue the distillation with pure water until the apparatus is free from traces of ammonia.

If the sample of water under examination contain much in the way of suspended matter, it will be wise to filter it before subjecting it to the ammonia process, through a filter that has been previously washed with the pure distilled water till free from ammonia. Any residual water having been syphoned out of the retort, 500 cc. of the clear sample are introduced, and a clean empty Nessler glass placed under the delivery tube of the condenser. Distillation is now proceeded with, and when 50 cc. of distillate have been collected in the Nessler glass, it is removed and an empty one put in its place. It now becomes necessary to ascertain how much free ammonia is present in the first 50 cc. of distillate; for this purpose run in 2 cc. of the Nessler solution from a pipette, well mix and place on a sheet of white paper. Observe the intensity of colour produced. If it is of a faint yellow tint there is not much in the way of "free "

ammonia present, but if it appears of a rich reddish brown colour a fair quantity is present, and sometimes when waters are highly contaminated with sewage, there is so much ammonia present as to occasion a brownish precipitate. In all such cases it is better to take a fraction of the 500 cc. for distillation, diluting to 500 cc. with the pure distilled water, previous to distillation. A burette graduated in 0.1 of a cc. is filled with the dilute standard ammonia solution, and a definite volume of it, say 3 cc., run into a clean Nessler glass; this is diluted with the pure distilled water to 50 cc., 2 cc. of Nessler solution added and well stirred; it is now placed beside the 50 cc. of distillate, and the colours compared by looking down through the liquids. If the solutions are not of equal depth of colour a fresh standard must be made, using more or less, as the case may demand, of the dilute ammonia solution, and Nesslerizing as before until a certain quantity used produces exactly the same colour as the sample. The "free" ammonia is estimated in the first 50 cc. of distillate only, $\frac{1}{3}$ of the quantity thus found being added on; this being the quantity that is invariably present in the next 150 cc. that are distilled over, before the introduction of the potash and permanganate solution for the determination of the albuminoid ammonia. Supposing that 2.5 cc. of the dilute standard ammonia solution were required for the first 50 cc. of distillate, that is, $2.5 \times .01 = .025$ milligram of ammonia, then the amount of free ammonia present in 500 cc. of sample will be $\frac{.025}{3} + .025 = .0333$ milligram, or $.0333 \times 2 = .0666$ parts per million, or $.0666 \times .07 = .00466$ grain per gallon.

The Albuminoid Ammonia.—After the 200 cc. have been distilled over and the free ammonia estimated as described, 50 cc. of the potash and permanganate are introduced into the retort, and the distillation proceeded with as before, Nesslerizing each 50 cc. that comes over, three of which are usually sufficient.

The following is an example of the determination of the free

and albuminoid ammonia in a sample of water contaminated with a little sewage :—

FREE AMMONIA.

500 cc taken.

1 cc. of standard dilute AmCl = .01 milligram of NH_3 .

1st 50 cc. distillation required 6.3 cc. = .063 milligram NH_3 .

Correction $\frac{1}{3}$ = .021 " "

Total . . .084

$\cdot 084 \times 2 = 0.168$ part per million,

or,

$\cdot 168 \times \cdot 07 = \cdot 01176$ grain per gallon.

ALBUMINOID AMMONIA.

1st 50 cc. required 5.0 cc. = .050 milligram NH_3 .

2nd " " 1.8 cc. = .018 " "

3rd " " 0.8 cc. = .008 " "

Total .076 " "

Subtracting NH_3 found in potash solution .005 " "

.071 " "

$\cdot 071 \times 2 = 0.142$ part per million,

or,

$0.142 \times \cdot 07 = \cdot 00994$ grain per gallon.

Remarks.—When the free ammonia in waters exceeds .08 part per million, it is often due to the fermentation of urea, into carbonate of ammonia, owing to the presence of urine, &c., in the water—the chlorine being likewise high; but there are waters which give a comparatively high proportion of free ammonia, with little albuminoid ammonia, and low chlorine. In such cases the free ammonia may be safely considered of saline or mineral origin rather than from a decomposition of organic pollution. When the albuminoid ammonia is high, and the free ammonia and chlorine low, it is diagnostic of the presence of organic matter of vegetable origin. In such cases the odour given off when the total solid residue is gently ignited,

will be of a peaty or some such characteristic vegetable odour. With regard to the amount of "free" and "albuminoid" ammonia present, that admits of a water being passed or condemned for drinking purposes, Professor Wanklyn states—

"If a water yield 0.00 part of albuminoid ammonia per million, it may be passed as organically pure despite of much free ammonia and chlorides; and if indeed the albuminoid ammonia amount to .02 or to less than 0.05 parts per million, the water belongs to the class of very pure water. When the albuminoid ammonia amounts to .05, then the proportion of free ammonia becomes an element in the calculation, and I should be inclined to regard with some suspicion a water yielding a considerable quantity of free ammonia along with more than 0.05 part of albuminoid ammonia per million. Free ammonia, however, being absent, or very small, a water should not be condemned unless the albuminoid ammonia reaches something like 0.10 part per million. Albuminoid ammonia above 0.10 per million begins to be a very suspicious sign; and over 0.15 ought to condemn a water absolutely. The absence of chlorine or the absence of more than 1 grain of chlorine per gallon, is a sign that the organic impurity is of vegetable rather than of animal origin; but it would be a great mistake to allow water highly contaminated with vegetable matter to be taken for domestic use."

The Nitrates.—The nitrogen present as nitrates and nitrites is to be determined as directed on page 156. Nitrates may be present in waters owing to the solvent action of the water on the nitrates present in the geological strata through which the water passes, or to the decomposition of nitrogenous organic matter present, with the production of nitric and nitrous acid, which in turn act upon the carbonates with the formation of nitrates and nitrites. Dr. Frankland lays much importance on the presence of nitrates in waters as indicating past pollution, and although the organic matter is thus destroyed, it may still contain living animalculæ capable of propagating disease; on the other hand, Dr. Wanklyn states

that "presence or abundance of nitrates does not show defilement by means of sewage, and deficiency of nitrates does not show absence of defilement." Whether or not the presence of nitrates is a measure of past or present organic pollution of a water is immaterial, since we have other means of determining its *actual* presence; and, moreover, knowing the filth that enters such rivers as the Thames every day previous to its distribution by the water companies for consumption, and that such waters are pronounced as being wholesome, it seems to be of secondary consideration what the condition of a water *was* so long as it comes up to the ideal of organic purity as it *is*.

The Nitrites.—A qualitative test for the presence of nitrites may be of interest in ascertaining whether any nitrogenous organic matter present is still decomposing, &c. The presence of nitrites may be quickly determined by the following method: 100 cc. of the water are taken, acidulated with 1 cc. of 5 E H_2SO_4 , 5 cc. of starch and potassic iodide solution (1 grm. of starch and 0.2 grm. of iodide in 100 cc.) added, and well stirred. If any considerable quantity of nitrites be present, a blue colour of iodide of starch will be at once produced, but if present only in minute quantity, the colour will not be produced until after standing for some time.

The Hardness.—The hardness may be determined as directed on page 150. It is always desirable to obtain *soft* water for laundry and toilet use, since it is more efficient and economic to use as a detergent than hard water, which would require artificial means for softening, such as boiling, or the use of excess of soap or soda. Where the supply of water is excessively hard, rain water, if it can be procured, would be far superior for such purposes. As a general rule, the hardness of a water increases with the total solids. It is said that persons who are predisposed to such diseases as *calculus* and *bronchocèle* are liable to propagate it if they habitually drink very hard water.

Poisonous Metals.—A drinking water should always be tested for lead. The presence of this metal is due to the solvent action of soft or faintly acid waters upon lead piping, which is often used to convey the water; and a test should likewise be made for copper and iron. A useful qualitative test for copper, lead, and iron collectively is as follows:—Pour 100 cc. of the water into a porcelain basin, add a drop of 5 E ammonium sulphide, and stir. If any copper, lead, and iron be present, a more or less dark colour will be produced, owing to the formation of the sulphides of the metals. If the colour produced is due to iron only, a drop of 5 E HCl will remove it, but will unaffected any sulphide of lead or copper present. If lead is detected by this means it should be determined by Dr. Miller's colorimetric method, as follows:—A standard solution of crystallized lead acetate is first prepared by dissolving 0.1831 gm. of the salt in a litre of distilled water, each cubic centimetre of such solution containing .0001 gm. of metallic lead; 70 cc. of the sample are poured into a white porcelain dish, two drops of 17 E acetic acid, and 5 cc. of a saturated solution of sulphuretted solution added, and stirred.

It now becomes necessary to imitate the test thus produced by a known volume of the standard lead solution; for this purpose pour about 65 cc. of distilled water into a similar porcelain dish, add 5 cc. of sulphuretted hydrogen water, stir, fill a burette with the standard lead solution, and allow it to run gradually into the distilled water, until the tests in both dishes are alike. Each cubic centimetre of lead solution required will represent $\frac{1}{16}$ of a grain of lead present in a gallon of water. Owing to the serious influence of water containing small quantities of lead on the human economy, all waters containing $\frac{1}{16}$ of a grain or upwards of the metal per gallon should be rejected for drinking purposes.

Copper.—This metal may be estimated in a manner similar to the above, the solution for comparison being prepared by dissolving 0.3929 gm. of crystallized sulphate of copper in a litre of water, each cc. containing .0001 gm. Cu.

Arsenic, Barium, Zinc, and Manganese, &c. sometimes find

their way into waters, especially in those mining and chemical works districts where the metals are obtained and worked.

The Colour of Waters.—Good drinking water should be free from colour. The colour of a sample of water can be best judged, by filling a colourless glass cylinder about two feet high with the sample and looking down through the liquid on to a sheet of white paper, making a comparison with distilled water contained in a similar cylinder.

The Suspended Matter.—The suspended matter if present in appreciable quantity, may be estimated as described on page 148, and it would be interesting to make, if possible, a microscopical search for any low forms of animal or vegetable life that it may contain.

Classification of Drinking Waters.—Dr. Frankland makes the following classification of waters for domestic purposes:—

Wholesome	{	1. Spring water	}	Very palatable.
		2. Deep well water		
Suspicious	{	3. Upland surface water	}	Moderately palatable.
		4. Stored rain water		
Dangerous	{	5. Surface water from cultivated land	}	Palatable.
		6. River water to which sewage gains access		
		7. Shallow well water		

The following is a classification of waters according to their softness:—

1. Rain water.
2. Upland surface water.
3. Surface water from cultivated land.
4. Polluted river water.
5. Spring water.
6. Deep well water.
7. Shallow well water.

THE POLLUTION OF RIVERS AND STREAMS.

Standards of Purity recommended by the Rivers Pollution Commissioners.—"We recommend that, with certain exceptions in reference to standards *d* and *e*, the following liquids be deemed polluting and inadmissible into any stream :—

"(a.) Any muddy liquid which has not been subjected to perfect rest in subsidence ponds of sufficient size for a period of at least six hours, *or* which, having been so subjected to subsidence, contains *in suspension* more than one part by weight of dry organic matter in 100,000 parts by weight of the liquid, *or* which, not having been subjected to subsidence, contains *in suspension* more than three parts by weight of dry mineral matter, or one part by weight of dry organic matter in 100,000 parts by weight of the liquid.

"(b.) Any liquid containing *in solution* more than two parts by weight of organic carbon, or 0·3 part by weight of organic nitrogen in 100,000 parts by weight.

"(c.) Any liquid which shall exhibit by daylight a distinct colour when a stratum of it one inch deep is placed in a white porcelain or earthenware vessel.

"(d.) Any liquid which contains, in solution in 100,000 parts by weight, more than two parts by weight of any metal except calcium, magnesium, potassium, and sodium.

"(e.) Any liquid which in 100,000 parts by weight contains, *whether in suspension or solution*, in chemical combination or otherwise, more than 0·05 part by weight of metallic arsenic.

"(f.) Any liquid which after acidification with sulphuric acid contains in 100,000 parts by weight more than one part by weight of free chlorine.

"(g.) Any liquid which contains in 100,000 parts by weight more than one part by weight of sulphur in the condition either of sulphuretted hydrogen or of a soluble sulphuret.

"(h.) Any liquid possessing an acidity greater than that which

is produced by adding two parts by weight of real muriatic acid to 1,000 parts by weight of distilled water.

“(i.) Any liquid possessing an alkalinity greater than that produced by adding one part by weight of dry caustic soda to 1,000 parts by weight of distilled water.

“(k.) Any liquid exhibiting a film of petroleum or hydrocarbon oil upon its surface, or containing, in suspension in 100,000 parts, more than 0.05 part of such oil.”

PART V.

OILS.

IN this section it is proposed to deal with oils in three divisions—(1) Oils for Lubricating, (2) Illuminating, (3) those adapted for special purposes. There is, of course, no hard and fast rule for the legitimate classification of oils in this manner, since some oils, such as rape, would come under the three divisions; but it will be found convenient.

(1) LUBRICATING OILS.

A lubricating oil has for its object the reduction of friction and the prevention of an undue development of heat. The result of friction is to abrade solids, heat being given out both in solids and liquids; the amount being the exact measure of the power wasted as a consequence of the friction. The amount of heat generated for each 772 foot pounds of work thus expended, is equivalent to the amount of heat necessary to raise the temperature of 1 lb. of water 1° F. Lubricants may be solid, semi-solid, or liquid, and the "body" or "viscosity" of them should be proportional to the pressure which will be exerted upon them. They may be fatty (animal or vegetable), mineral, or compound. Soap is a constituent of railway grease, and graphite and steatite are sometimes used for heavy machinery.

The following list shows the best purpose to which the various lubricants can be applied :—

For steam cylinders . . .	{ Heavy mineral oils, lard, tallow, rape oil, &c.
Ordinary machinery . . .	{ Rape oil, lard oil, tallow oil, and medium mineral oils.
For very great pressures with slow speed . . .	{ Graphite, soapstone, &c.
For great pressures with slow speed . . .	{ Tallow, lard, palm oil, grease, &c.
For heavy pressures and high speeds . . .	{ Sperm oil, rape oil, castor oil, medium mineral oils.
For light pressures and high speed	{ Sperm, refined petroleums, cottonseed, rape, olive, and mineral oils.
Watches, clocks, &c. . .	{ Light mineral oils, clarified sperm, neats-foot, olive and porpoise.

It may be stated, however, that in all cases where possible, a mineral oil of suitable "body" should be used, especially for cylinders, the high temperature of which decomposes fatty oils, with the production of free fatty acids, which attack copper, iron, &c., forming metallic soaps, which cause much damage to fittings, &c.

In judging of the suitability of a lubricating oil, the purpose for which it is proposed to be used being first ascertained, the following points must be determined :—

(1) The "body" or viscosity of the sample at the temperature at which it is proposed to be used.

(2) The specific gravity at 15° C., or in some cases at the temperature of boiling water.

(3) The "flashing point," or the temperature at which the vapour evolved becomes ignited on the application of a flame.

(4) The percentage of free, fatty, or mineral acids present.

(5) The percentage of fatty and mineral oils.

(6) The loss the oil suffers when exposed for about twelve hours to the temperature at which it is proposed to work.

- (7) The amount, if any, of mineral and organic suspended matters present.
- (8) The temperature at which the oil solidifies or congeals.
- (9) A test to ascertain the liability of the oil to gum.
- (10) The extent of the corroding action of the oil on pieces of steel, brass, bronze, &c.

Determination of Viscosity.—The viscosity or body of an oil, although not an exact measure of unctuousness or greasiness, is one of the most important factors in judging of its suitability for any special class of work. The viscosity should be proportional to the pressure exerted upon the oil—that is to say, an oil for any specific purpose should not be so thick as to cause undue fluid friction, nor so thin that it is squeezed out, leaving the bearings, &c., dry.

The viscosity of an oil decreases considerably with an increase in temperature; mineral oils in this respect decreasing, through a given range of temperature, far more than the majority of fatty oils. There is no fixed ratio between the viscosities of oils at the ordinary temperature, and the viscosities at an elevated temperature, *i.e.*, two different oils having identical viscosities at 15° C. would in all probability have different viscosities at a temperature, say, of 100° C. The specific gravity of an oil, although valuable as a means in some cases of identification, is no criterion of the viscosity. The best form of apparatus suitable for obtaining viscosities at different temperatures, is that devised by Mr. Boverton Redwood, F.R.S.E., &c. Each apparatus is standardized by Mr. Redwood himself, being so adjusted that 50 cc. of a standard average rape oil take 535 seconds to flow out at a temperature of 15.5° C., distilled water taking 25.5 seconds to flow out under the same conditions. The following is a sketch (Figs. 21 and 22) of the apparatus* and *modus operandi* of the process:—

The viscosity tube A is of electro-plated copper 3½ inches

* The standardized apparatus can be obtained from Townson & Mercer, 89, Bishopsgate Street, E.C., and James How & Co., 73, Farringdon Street, E.C.

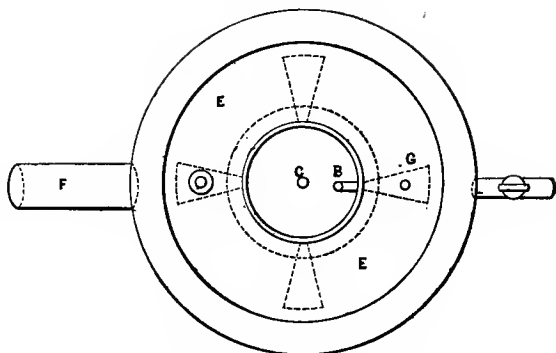


FIG. 21.—Horizontal Section.

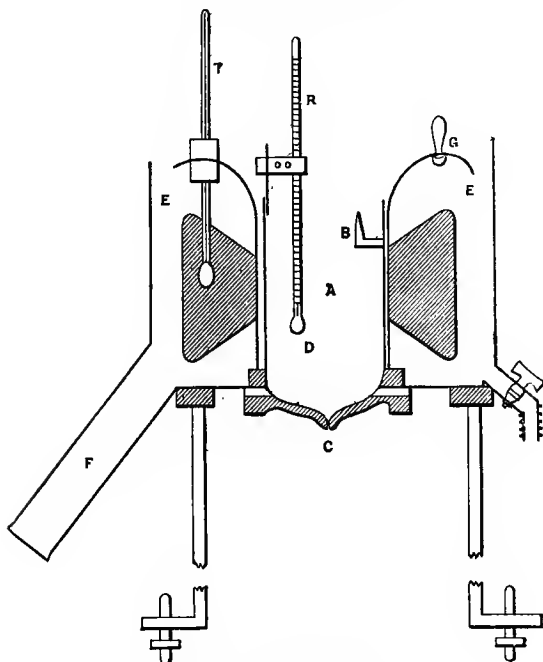


FIG. 22.—Vertical Section.

REDWOOD'S APPARATUS FOR DETERMINING VISCOSITY OF OIL

high and $1\frac{1}{8}$ -inch internal diameter. The orifice *c* is of agate, of definite bore and limited protuberance, and a plug of electroplated copper suspended to a wire is supplied with the apparatus, in order to close it while the oil is being run in and adjusted. The outer jacket *e* is made of copper, in which water, oil, or any other liquid can be maintained at a definite temperature by the application of a flame to *f*. The temperature is made uniform by means of the paddles which can be revolved with the handle *g*.

In order to obtain viscosities at elevated temperatures, *e* is filled with mineral oil of high-boiling point, and heated with a Bunsen until the desired temperature is attained, which is kept constant by the regulation of the flame, and the oil mixed by means of the paddles. The sample of oil to be tested is heated in an air bath to the required temperature, and poured into the viscosity tube *A* until the surface of the oil just reaches the point of the gauge *B*. When it is assured that the temperatures registered by the thermometers *T* and *R* are the same, a small narrow-necked flask holding 50 cc. to a mark made on its neck, is placed under the orifice in a vessel containing a liquid heated to the same temperature as the oils. The plug is then taken out of *c*, and the number of seconds that 50 cc. of the oil take to run out, is noted.

The temperature at which the viscosity of an oil should be determined, depends upon the temperature at which it is proposed to be used. With oils proposed to be used for ordinary machinery, the viscosities should be determined at temperatures of 15° , 30° , and 60° C.; while for cylinders, at temperatures of 100° , 120° , and 150° C.

The following interesting table by Redwood,* gives the comparative rates of flow of various oils for every rise of 10° F., by the above standard apparatus:—

* *Vide* "Journal Soc. Chem. Ind.," v. 128.

Temperature °F.	Refined Rape Oil.	Refined Rape Oil.	Refined Rape Oil.	Refined Rape Oil.	Beef Tallow.	Sperm Oil.	Neatsfoot Oil.	American Mineral Oil. Sp. gr. '885.	American Mineral Oil. Sp. gr. '913.	American Mineral Oil. Sp. gr. '923.	Russian Mineral Oil. Sp. gr. '909.	Russian Mineral Oil. Sp. gr. '915.	Russian Mineral Oil. Semi-solid.
50	712.5	—	—	—	—	—	620	145	425	1030	2040	2520	—
60	540	—	—	—	—	177	470	105	295.5	680	1235	1980	—
70	405	406	405.5	407	—	137	366	90	225	485	820	1320	—
80	326	—	—	—	—	113	280	73	171	375	580	900	—
90	260	—	—	—	—	96	219	63.5	136	262	426	640	—
100	213.5	—	—	—	—	80.5	175	54	111	200	315	440	1015
110	169	—	—	—	—	70.5	147.5	50	89.5	153	226	335	739.5
120	147	146	147	147.5	—	60.5	126	47	78	126	174	245	531
130	123.5	—	—	—	—	57	112	44.75	63.5	101	135.5	185	398.5
140	105.5	106.5	106.5	106	—	54	88.5	41	58	82	116	145	317.5
150	95.5	—	—	—	—	49	75.5	37.5	52	70.5	95	115	250
160	85	—	—	—	—	47.5	70	—	46	63.5	83.5	93.5	200
170	76	—	—	—	—	46	62	—	—	58	70.5	77.5	161
180	69	—	—	—	—	44.5	56.5	—	—	52.5	61.5	67.5	134.5
190	64.5	—	—	—	—	43	53	—	—	47	56.5	61	115.5
200	58.5	57.5	57.5	58.5	54.5	42	50.5	—	—	42	48	54	99
210	54	—	—	—	—	41	48.5	—	—	40	—	—	85
220	54	—	—	—	—	39	47	—	—	38	—	—	77
230	47.5	—	—	—	—	37	46	—	—	—	—	—	70.5
240	45.5	—	—	—	—	36	44.5	—	—	—	—	—	64.5
250	43.5	—	—	—	40	35	44	—	—	—	—	—	59
260	—	—	—	—	—	34	43.5	—	—	—	—	—	54
270	—	—	—	—	—	33	43	—	—	—	—	—	48.5
280	—	—	—	—	—	32	41.5	—	—	—	—	—	46.5
290	—	—	—	—	—	31	41	—	—	—	—	—	46
300	—	—	—	—	—	30	38	—	—	—	—	—	42.5

The following table gives the number of seconds that 50 cc. of oil (mineral) take to run out of Redwood's Viscometer at various temperatures, and the purposes for which such oils the writer has found to be most suitable:—

	At 60° F.	140° F.	180° F.	220° F.
	Secs.	Secs.	Secs.	Secs.
Cylinders, valves, and heavy machinery	—	—	95	65
Medium machinery, carriage oils, boxes, &c.	540	105	70	54
Light machinery, spindles, &c.	290	52	30	15

Specific Gravity.—The specific gravity of oils is usually determined at a temperature of 15°C ., and in the case of solid fats, at any convenient temperature above their melting points. A quick and fairly accurate specific gravity of oils fluid at the ordinary temperature, may be obtained by the hydrometer (Fig. 23). The oil having been brought to the required temperature in a glass cylinder, the hydrometer is dropped into it; when it stops sinking, the point of the graduated stem, which the tip of the meniscus of the oil just touches, is taken as the specific gravity. In the examination of oils, it will be convenient to keep three hydrometers, graduated from .700 to .800, .800 to .900, and .900 to 1.000 respectively. Some oils are too thick to allow their specific gravities being taken with the hydrometer at the ordinary temperature; in such cases the specific gravity bottle (Fig. 24) may be used—one holding about 50 cc., with a fine hole drilled through the stopper is convenient. The weight of the empty dry bottle being first ascertained, it is filled with distilled water at 15°C ., and again weighed; and by subtracting the weight of the bottle, the weight of the water it holds is thus obtained. The water having been poured away and the bottle dried, the sample of oil at a temperature of 15°C . is poured in, and the bottle

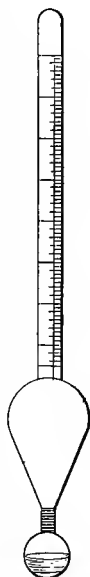


FIG. 23.



FIG. 24.

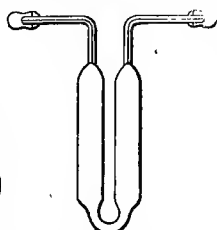


FIG. 25.

stoppered, cleaned, dried and weighed. The weight of the oil thus obtained (by subtracting the weight of the bottle from that of the bottle plus oil), divided by the weight of water the bottle holds, will give the specific gravity of the oil.

In determining the specific gravity of the solid fats at elevated temperatures, Sprengel's specific gravity tube (Fig. 25)

is useful. The weight of water at 15° C. it holds having been first ascertained, the melted fat is sucked into it and placed into a beaker of water at the desired temperature; after being immersed sufficiently long for the oil to acquire the temperature of the water, the caps are put on each of the ends of the capillary tubes, dried, cooled and weighed, from which the specific gravity is calculated; water at 15° C. being taken as unity. It sometimes happens that in extracting solid fats, &c., from substances, there is insufficient to make a determination of the specific gravity by the usual methods; in such cases it can be obtained fairly accurately by floating a thin piece of the fat on distilled water at the desired temperature, and adding alcohol, until on moving the fat about with a glass rod, it will stop in any desired part of the liquid. The specific gravity of the liquid being ascertained with the hydrometer or bottle, the specific gravity of the fat is obtained.

The following is a table of specific gravities, at 15° C., by Stillwell, of various kinds of animal and vegetable oils:—

Sperm, bleached winter8813
Sperm, natural winter8815
Elaine9011
Red, saponified9016
Palm9046
Tallow9137
Neatsfoot9142
Rape-seed, white winter9144
Olive, light greenish yellow9144
Olive, dark green9145
Pea-nut9154
Olive, virgin, very light yellow9163
Rape-seed, dark yellow9168
Olive, virgin, dark clear yellow9169
Lard, winter9175
Sea elephant9199
Tanner's (Cod)9205
Cotton-seed, raw9224
Cotton-seed, refined yellow9230
Salad (cotton-seed)9231
Labrador (Cod)9237
Poppy9245

Seal (natural)	°246
Cocoa nut	°250
Whale, natural winter	°254
Whale, bleached winter	°258
Cod liver, pure	°270
Seal, raked	°286
Cotton-seed, white winter	°288
Straits (Cod)	°290
Menhaden (dark)	°292
Linseed (raw)	°299
Bank (cod)	°320
Menhaden (light)	°325
Porgy	°332
Linseed, boiled	°411
Castor, pure, cold pressed	°667
Resin, third run	°887

The mineral lubricating oils, have specific gravities ranging from °850 to °925.

The following is a table of specific gravities and yield of various products on the distillation of 100 gallons of Russian petroleum :—

	Specific Gravity.	Gallons.
"Benzine"	°725	1
"Gasolene"	°775	3
Kerosene	°822	27
Pyronaphtha	°858	12
Lubricating oil	°890-°905	27
Cylinder oil	°915	5
Vaseline (not a direct product)	°925	1
Residuum and loss	—	24
		<hr/> 100

The Flashing Point.—The flashing point, or the temperature at which an oil gives off inflammable vapour, can be determined by the Abel flash-point apparatus, described on page 204; but the cup must be taken out of the water bath and placed in a copper air bath, since the flash point of most lubricating oils is above the temperature of boiling water. A small flame is kept under the apparatus, and the oil tested at every rise of one degree, as in the case of the petroleums. The flash point

of a lubricating oil must be higher than the temperature at which it is proposed to work. For cylinders, &c., oils should not flash below 200° C., and for ordinary machinery not below 149° C. The light Scotch shale oils flash at from 130° to 185° C., while the darker variety used for heavy machinery, cylinders, &c., flash at from 180° to 230° C.

Determination of Free Fatty and Mineral Acids.—

Free fatty acids present in fatty oils are brought about by the decomposition of the glycerides, which thus become rancid. The free fatty acids, present in oils intended for lubricating, should not exceed 5 per cent., owing to their corroding action on bearings, &c.; while for burning purposes, 3 per cent. should be the maximum quantity present, otherwise the wicks become charred. Free mineral acids are sometimes present in very small quantities, owing to their imperfect elimination during refining; they have a very corroding action on bearings, &c. The free fatty acids may be determined as follows: Weigh out 50 grms. of the sample (or in the case of palm oil, 5 grms.) into a 200 cc. flask, and pour on to it 100 cc. of neutral methylated alcohol, to which three drops of an alcoholic solution of phenolphthalein (1 in 30), and one drop of E NaHO has been previously added; the pink solution thus produced soon becomes colourless on agitating with the oil if any free fatty acid be present. After agitating for some time to ensure the complete solution of the free acids, a *standard* E solution of sodic hydrate is gradually run in from a burette, stirring, until the last drop added produces a permanent pink coloration on agitating for five minutes. Note the volume of standard soda solution thus required. Each cc. of solution required, corresponds to 0.282 gm. of free oleic acid ($C_{18}H_{34}O_2$), 0.256 gm. free palmitic acid ($C_{16}H_{32}O_2$), or 0.284 gm. of free stearic acid ($C_{18}H_{36}O_2$), as the case may be.

Free mineral acids may be detected by agitating 50 cc. of the oil with about 30 cc. of distilled water free from acid; and on adding an alcoholic solution of methyl-orange to the aqueous extract, a more or less pink coloration will be pro-

duced if any free mineral acid be present. An idea of the amount could be ascertained by tritating with standard $\frac{E}{10}$ NaHO. The nature of the free mineral acids could be determined by dividing an aqueous extract of 50 cc. of the sample into three equal parts, adding $BaCl_2$ to one for H_2SO_4 , $AgNO_3$ to another for HCl , and sulphate of brucine, and 36 E H_2SO_4 to another for HNO_3 . When free mineral acid is present in oils it is mostly sulphuric acid. A very delicate test for the detection of minute quantities of free sulphuric acid is to evaporate an aqueous extract of about 20 grms. of the oil, to dryness, with an addition of a few crystals of white cane sugar, and on heating the residue for some time in a water bath, a more or less browning or blackening will be produced.

The following table by Watson shows the corroding action of various oils upon iron and copper ;—

Oils.	Iron dissolved in 24 days.	Copper dissolved in 10 days.
Almond	·0040 grain	·1030
Castor	·0048 "	—
Colza	·0800 "	·0170
Lard	·0250 "	—
Linseed	·0050 "	·3000
Neatsfoot	·0875 "	·1100
Olive	·0062 "	·2200
Paraffin	·0045 "	·0015
Seal	·0050 "	·0485
Sperm	·0460 "	·0030

The extent of the action of oils on metals, is directly proportional to the amount of free fatty acids present, and most of the fatty oils act more rapidly upon copper than on iron.

The following table * shows the minimum, maximum, and mean percentage proportions of free fatty acids in various oils ;—

* Noerdlinger, "Zeit. Anal. Chem.," 28, 183-187.

- A. Table oils obtained by pressing.
 B. Expressed commercial oils.
 C. Oils extracted by light petroleum.

	A.			B.			C.		
	min.	max.	mean	min.	max.	mean.	min.	max.	mean.
Rape-seed oil . . .	0.53	1.82	1.19	0.52	6.26	2.88	0.77	1.10	0.93
Olive oil . . .	—	—	1.66	3.87	27.16	12.97	—	—	—
Poppy oil . . .	0.70	2.86	1.92	12.87	17.73	15.37	2.15	9.43	4.72
Earth-nut oil . . .	0.85	3.91	1.94	3.58	10.61	6.52	0.95	8.85	4.02
Oil of sesame . . .	0.47	5.75	1.97	7.17	33.13	17.94	2.62	9.71	4.89
Cotton-seed oil . . .	—	—	0.15	0.42	0.50	0.46	—	—	—
Oil of mustard . . .	—	—	—	0.68	1.02	0.85	—	—	—
Castor oil . . .	—	—	—	0.62	18.61	9.28	1.18	5.52	2.78
Linseed oil . . .	—	—	—	0.41	4.19	1.57	—	—	—
Candle-nut oil . . .	—	—	—	—	—	56.45	—	—	—
Palm kernel oil . . .	—	—	—	3.3	17.65	6.91	4.17	11.42	8.49
Palm oil (old) . . .	—	—	—	—	—	50.82	—	—	—
Cocanut oil . . .	—	—	—	3.03	14.35	7.92	1.00	6.31	4.26
Llippe fat (3 years old)	—	—	—	—	—	28.54	—	—	—
Niam fat . . .	—	—	—	—	—	—	14.40	34.72	24.56
Bicuhyba fat . . .	—	—	—	—	—	18.55	—	—	—
Japan wax . . .	—	—	—	—	—	9.25	—	—	—

Determination of Fatty and Mineral Oils.—Mixtures of fatty and mineral oils are now much used in lubrication, and it becomes frequently necessary to determine the proportion of each present. In order to effect their separation, advantage is taken of the fact that, while fatty oils are decomposed and converted into soaps by the action of a solution of potash or soda, the mineral oils do not suffer any apparent change by such treatment. The following is the *modus operandi* of the process :—

Weigh out 5 grms. of the sample into a 5-oz. wide-mouth stout glass bottle, provided with an indiarubber stopper. Pour into it 26 cc. of $1\frac{1}{2}$ E alcoholic potassic hydrate solution, and place into a bath of cold water. A Bunsen is lit under the bath, and when the alcoholic solution is seen to boil, the bottle is taken out, and the stopper fixed and tied down with wire; it is now again placed into the bath, the water made to

boil, and the bottle occasionally shaken ; after heating thus for about half an hour, the bottle is taken out, allowed to cool, the contents poured into a porcelain basin, and evaporated nearly to dryness over a water bath ; 60 cc. of warm water are now poured into the bottle and well shaken in small quantities at a time, so as to remove any adhering matter, poured into the dish, and stirred with a glass rod until the soap goes into solution : any mineral oil present will float, or remain in an emulsive condition with the solution of soap. The whole is now carefully poured into a globular or cylindrical separator (Figs. 26 and 27), and the dish and bottle washed out with water and about 50 cc. of methylated ether (using small quantities at a time), into the separator ; the stopper is now put in, the contents violently shaken up for about ten minutes, and the mixture allowed to separate ; sometimes there is a little difficulty in the separation, an emulsion being formed. In such cases an addition of 2 or 3 cc. of absolute alcohol with a rotatory motion puts matters all right. When complete separation is ensured, the aqueous solution is tapped off into a second separator, the aqueous solution shaken up with 20 cc. more ether, allowed to separate, the aqueous solution tapped off as before, and the ethereal extract added to the main portion, which in turn is washed three times with 20 cc. of water, and the washings added to the main aqueous solution. The ethereal solution of the mineral oil is now tapped off into a weighed flask attached to a Liebig's condenser, and the ether distilled off ; the residual mineral oil is then dried in a water oven, air being pumped into it occasionally to get rid of the last traces of water, which was taken up by the ether ; after which it is cooled and weighed, and dried again until no further loss occurs. If the mineral oil present be light, and of low boiling point, considerable loss would be incurred by its evaporation by this process ;



FIG. 26.

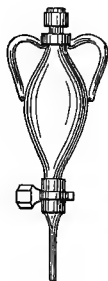


FIG. 27.

in such cases it would be best to determine by difference, or dry at a lower temperature where practicable. The specific gravity, congealing, and melting points, would convey an idea of the nature and source of the mineral oil present. The aqueous solution contained in a separator, which now contains the oil in the form of soap, is acidulated with 10 cc. of 10 E HCl, the fatty acids being thus precipitated. 40 cc. of methylated ether are added, and the mixture well shaken up; the fatty acids are thus dissolved by the ether. After complete separation occurs, the acid solution is tapped off, and the ethereal solution washed three times with about 20 cc. of water, after which it is run off into a weighed flask, the separator being washed out with a little ether. The flask is attached to a Liebig's condenser, the ether distilled off, and the residual fatty acids dried in the water oven until no further loss of weight occurs. On adding 5 per cent. for glycerine, to the weight of the fatty acids thus obtained, this gives practically the proportion of fatty oil present. In order to identify the oil, obtain its specific gravity, melting point, odour on ignition, and if resin is indicated estimate by Gladding's process, given on page 237.

The following table by Hübl gives the melting and congealing points of fatty acids from various oils and fats, and will serve as a good guide as to the nature of the oil present:—

Fatty Acids from—	Melting point. °C.	Congeaing point. °C.
Olive oil	26	21·2
Almond oil	14	5·0
Arachis oil	27·7	23·8
Rape oil	20·1	12·2
Cottonseed oil	37·7	30·5
Sesamé oil	26·0	22·3
Linseed oil	17·0	13·3
Poppy oil	20·5	16·5
Hempseed oil	19·0	15·0
Walnut oil	20·0	16·5
Castor oil	13·0	3·0
Palm oil	47·8	42·7
Cacao butter	52·0	51·0

Fatty Acids from—	Melting point. °C.	Congeaing point. °C.
Nutmeg butter	42·5	40·0
Cocconut oil	24·6	20·4
Laurel oil	27·0	22·0
Shea butter	39·5	38·0
Tallow	45·0	43·50
Wool fat	41·8	40·0
Butterine	42	39·8

Determination of the Melting and Congealing Points of Oils and Fats.—It is important that oils used for lubrication should not have very high congealing or solidifying points. The temperature at which an oil congeals may be found by half filling a test tube, 6 in. by $\frac{1}{2}$ in., with the oil, placing a thermometer therein, and introducing the whole into a mixture of equal parts of salt and ice; or if ice be not available, into a mixture of fifteen parts of sodic sulphate, and ten parts of 5 E HCl. The oil is stirred with the thermometer until on inclining the test tube the oil ceases to run; the temperature at which this takes place is taken as the congealing point. On taking the test tube out of the mixture the temperature at which the oil melts could be obtained. It is important that the sample should be free from water and suspended matter before obtaining the melting or congealing point, since these interfere with the accuracy of the results. To obtain the melting and congealing points of oils, solid or semi-solid at the ordinary temperature, a small tall beaker is nearly filled with mercury, and placed in a bath of cold water, the surrounding water being slightly higher than the level of the mercury. A thermometer is suspended from a support, the bulb of which is immersed in the beaker of mercury; a little of the fat—about the size of a pin's head—is placed on the mercury by means of a pair of glass rods drawn out to fine points, and a small flame placed under the water bath, regulated so that the temperature of the mercury rises about $0\cdot5^{\circ}$ C. per minute. Note the temperature at which the sample begins to melt, and also the temperature at which it is perfectly fluid. By taking the

lamp away it could be observed, as the temperature sinks, at what point the sample congeals.

Good mineral oils do not congeal at the freezing point of water; the heavier varieties congeal at about -6° to -8° C., and the lighter kinds remaining fluid at -16° to -18° C.

The following results were obtained at the U.S. Navy Yard, Brooklyn, in 1870, with various oils:—

	Thickens. ° C.	Flow ceases. ° C.	Solid. ° C.	Sp. gr.
Sperm . . .	1	— 3	— 9	·761
Olive . . .	10	— 8	— 14	·933
Tallow . . .	— 1	— 4	— 8	·795
„ . . .	21	16	6	·993
Lard . . .	7	0	— 6	·959

The following are congealing points of the more important oils:—

	° C.
Rape oil, about	— 8
Cottonseed oil, about	— 3
Sunflower „ „	— 14
Linseed „ „	— 24
Hempseed „ „	— 20
Castor „ „	— 18
Poppyseed „ „	— 18
Neatsfoot „ „	— 4

The following are melting points of a few fats, solid at the ordinary temperature:—

	° C.
Cacao butter, about	32
Palm oil „	35
Cocoanut oil „	26
Tallow „	43
Butter „	34
Butterine „	38
Beeswax „	63
Spermaceti „	46

Loss on Evaporation, and Gumming Properties of Oils.—To determine the loss an oil sustains on exposure at

elevated temperatures, weigh out about 1 grm. of the oil on a weighed watch glass, allow to stand exposed in a water oven for twelve hours, cool and reweigh, and calculate the loss or gain, as the case may be; observe if the residue is at all sticky or gummy. If the oil be required for light machinery, exposure for twelve hours at 60° C. instead of 100° will be found suitable. Good mineral oils, intended for heavy machinery, cylinders, &c., rarely lose more than 1 per cent. on exposure for twelve hours at 100° C., while mineral oils intended for light machinery rarely exceed a loss of 0.5 per cent. on exposure for twelve hours at 60° C. Many vegetable oils of the "drying" class, such as linseed, hempseed, cottonseed, &c., increase in weight on exposure for twelve hours at 100° C., owing to the absorption of oxygen from the air.

Estimation of Suspended Matter.—Oils intended for lubrication should only contain, at most, a mere trace of suspended matter. Some mineral oils are so dark and thick that a fair quantity of suspended or mineral matter would escape observation in the ordinary way. To determine the proportion that may be present, weigh out about 10 grms. of the sample into a beaker, dilute with 50 cc. of methylated ether, filter off any suspended matter through a weighed filter paper, wash the filter till free from oil with ether, dry and weigh. Increase = suspended matter both mineral and organic. If the amount be large, it would be wise to ignite the filter and its contents carefully in a weighed crucible, and estimate the residual mineral matter. The loss on the former weight would represent suspended organic matter.

The following should be the ideal characteristics of good lubricating oils, according to Spon:—

- (1) A mineral oil flashing below 150° C. is unsafe.
- (2) A mineral oil losing more than 5 per cent. in ten hours at 15° to 20° C. is inadmissible, as the evaporation creates a gum, or leaves the bearing dry.
- (3) The most fluid oil, that will remain in its place, fulfilling

other conditions, is the best for all light bearings at high speeds.

(4) The best oil is that which has the greatest adhesion to metallic surfaces, and the least cohesion in its own particles; in this respect fine mineral oils stand first, sperm oils second, neatsfoot oil third, and lard oil fourth; consequently the finest mineral oils are best for light bearings and high velocities. The best animal oil to give body to fine mineral oils is sperm oil; lard and neatsfoot oils may replace sperm oil when greater tenacity is required.

(5) The best mineral oil for steam cylinders is one having a density of $\cdot 893$, and a flashing point of 360° C.

(6) The best mineral oil for heavy machinery has a density of $\cdot 880$, and a flashing point of 269° C.

(7) The best mineral oil for light bearings and high velocities has a density of $\cdot 871$, and a flashing point of 262° C.

(8) Mineral oils alone are not suited for very heavy machinery, on account of their want of body, but well purified animal oils are applicable to the heaviest machinery.

(9) Olive oil stands first among vegetable oils, as it can be purified without the aid of mineral acids. The other vegetable oils which, though far inferior to olive oil, are admissible as lubricants are—in their order of merit—sesamé, earth-nut, rape and colza, and cottonseed oils.

(10) No oil is admissible which has been purified by means of mineral acids.

The following list of oils, by Duggan, are placed in the order of their usual commercial monetary value :—

- | | | |
|-----|---|---------------------|
| (1) | | Olive oil. |
| (2) | | Sperm oil. |
| (3) | | Neatsfoot oil. |
| (4) | { | (a) Bottlenose oil. |
| | | (b) Lard oil. |
| | | (c) Castor oil. |
| (7) | | Cod oil. |
| (8) | { | (a) Arachis oil. |
| | | (b) Sesamé oil. |
| | | (c) Poppyseed oil. |

(11)	Colza and Rape oil.
(12)	Seal oil.
(13)	Nigerseed oil.
(14)	Linseed oil.
(15)	Whale oil.
(16)	Cottonseed oil.
(17)	Menhaden oil.
(18)	Japan Fish oil.
(19)	Mineral oils.
(20)	Resin oil.

The following is a useful table, by Allen, for the examination of oils containing foreign admixtures :—

SCHEME FOR ANALYSING OILS CONTAINING FOREIGN ADMIXTURES.

From 5 to 10 grms. of the sample (previously melted if necessary) is passed through a filter, unless clear.

Residue may contain *salt, curd, sand,* and insoluble matter generally. It may be washed with ether, dried, and weighed; then ignited, and weighed again, the difference being the organic matter.

Aqueous liquid may contain light metals. It is evaporated to dryness at 100° C., and the residue weighed and further analysed if desired.

Acid liquid may contain sulphates of aluminium and heavy metals previously existent as soaps.

Oil solution. Agitate with 5 E sulphuric acid and separate. Wash residual oil repeatedly by agitation with water till the aqueous liquid no longer reddens litmus paper.

Solution of oil in ether or carbon disulphide. Distil off the solvent and beat the residual oil with alcohol and a few drops of solution of phenolphthalein. Then add *standard* E soda cautiously, agitating the whole between each addition until a pink colour is obtained, which remains after shaking. Note the volume of soda solution required, as the quantity used is a measure of the free, fatty, and resin acids, and will determine the necessity of looking further into their nature. Separate any undissolved oil, dilute the alcoholic liquid, evaporate off the alcohol at a gentle heat and agitate with petroleum spirit, separate, evaporate off solvent, and add any separated oil to the main portion.

Aqueous liquid. Add 10 E HCl and agitate with ether or carbon disulphide; evaporate the ethereal layer at 100°, and weigh residue, representing mixed, free, fatty, and resin acids of original sample, and if soaps of aluminium or heavy metals were present, of the fatty acids resulting from their decomposition.

Oil. Saponify with $\frac{1}{2}$ E alcoholic potash solution. Boil off alcohol, dissolve soap in warm water, and agitate cooled solution with ether. Separate and agitate aqueous liquid a second and third time with ether.

Aqueous liquid contains the glycerine and the soap formed by saponification of the neutral fixed oil of the sample. If the solution be treated with 10 E HCl and agitated with ether, the weight of the fatty acids left on evaporating the ethereal layer, multiplied by 1.055 will give approximately the weight of the neutral fixed oil.

Ethereal liquid distilled at 100° C., and the last traces of the solvent removed by a current of air or other means, leaves the hydrocarbon oils in a state fit for identification and further examination.

MINERAL ILLUMINATING OILS.

The mineral illuminating oils of commerce are obtained by distilling (1) The natural crude petroleum oils found as springs and subterranean reservoirs in close proximity to deposits of bitumen, bituminous coals, &c., in America, South Russia, India, Persia, &c., and (2) The crude oil obtained by the distillation of bituminous shale.

The oil fields of Pennsylvania supply most of the burning oils of commerce.

There are many fancy names given to the various fractions of the distillation of crude petroleum and shale oils sold as burning oils, such as "kerosene," "photogene," "petroline," "liquid gas," "beacon oil," "aurora oil," "astral oil," &c. The following table by Redwood* shows the percentage of burning oils obtained from crude petroleums of various localities:—

Locality.	Specific gravity.	Yield of Commercial Products.			
		Naphtba	Burning oil.		Lubrica- ting oil.
		per cent	per cent	sp. gr.	per cent.
Persia	·777	1.4	87.5	—	—
East India	·821	3.6	62.5	·800	32.0
Burmah, mud volcano, Kyouk Phyou	·818	nil	55.7	·800	31.3
„ native pits, Mimbyin . .	·866	„	15.1	·810	65.9
„ Western Barangah . . .	·888	„	7.2	·815	89.3
„ Eastern Barangah . . .	·835	2.5	66.1	·810	27.3
Assam	·933	nil	nil	—	94.2
India	·935	„	20.0	·805	60.0
Russia	·836	20.0	40.0	—	37.5
„	·942	nil	nil	—	90.0
Hanover	·843	10.0	60.0	·812	27.5
South America	·852	nil	50.0	·808	45.0
„	·900	„	nil	—	91.5
New Zealand	·828	„	60.0	·808	38.0
Italy, near Milan	·787	45.0	45.0	·806	5.4
United States, Wyoming . .	·910	2.5	27.5	—	57.5
„ „	·945	nil	10.0	—	72.5
Scotch Shale Oil	·870	6	36	·810	18.0

* "Journ. Soc. Arts," xxxiv., 823, 878.

Assay of Burning Oils.—One of the most important points to be determined in the selection of mineral oils for burning purposes is the temperature at which the vapour evolved from the oil takes fire on the application of a flame, and is termed the “flashing point.” The minimum flashing point by the close test about to be described, has been fixed by the Petroleum Act of 1879 to be 73 degrees of Fahrenheit’s thermometer. The following is a sketch of the apparatus and *modus operandi* of the process for the determination of the flashing points of light mineral oils as devised by Sir Frederick Abel, and set forth in Schedule I. of the Petroleum Amendment Act of 1879:—

Specification of the Test Apparatus (Fig. 28).—The oil cup consists of a cylindrical vessel 2 in. in diameter and $2\frac{2}{5}$ in. in height (internal), with outward projecting rim $\frac{1}{10}$ in. wide, $\frac{3}{8}$ in. from the top and $1\frac{7}{8}$ in. from the bottom of the cup. It is made of gun-metal or brass (17 B.W.G.) tinned inside. A bracket consisting of a short stout piece of wire bent upwards, and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is $1\frac{1}{2}$ in. The cup is provided with a close-fitting overlapping cover made of brass (22 B.W.G.), which holds the thermometer and test lamp. The latter is suspended from two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, the mouth of which is $\frac{1}{8}$ in. in diameter. The socket which is to hold the thermometer is fixed at such an angle, and its length so adjusted, that the bulb of the thermometer when inserted to its full depth shall be $1\frac{1}{2}$ in. below the centre of the lid.

The cover is provided with three square holes, one in the centre $\frac{1}{10}$ in. \times $\frac{1}{10}$ in., and two smaller ones $\frac{3}{16}$ in. \times $\frac{3}{16}$ in. close to the sides and opposite each other. These three holes may be closed and uncovered by means of a slide moving in grooves, and having perforations corresponding to those on the lid.

In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such a way as to bring the end of the spout just below the surface of

the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position.

Upon the cover, in front of and in line with the mouth of the lamp, is fixed a white bead, the dimensions of which represent the size of the test flame to be used.

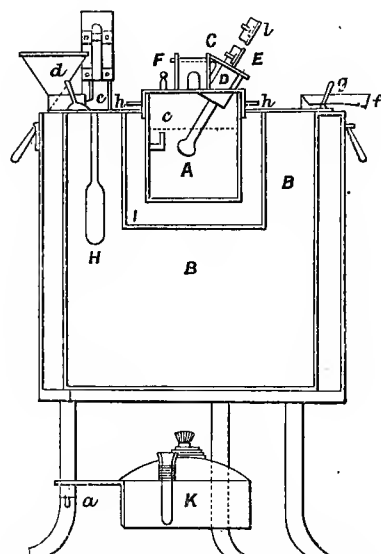


FIG. 28.—SIR F. ABEL'S FLASH-POINT APPARATUS.

- | | |
|---------------------|----------------|
| B Water bath. | I Air chamber. |
| A Oil cup. | H Thermometer. |
| C Gauge. | K Spirit lamp. |
| E Thermometer. | F Flame gauge. |
| G Oscillating lamp. | d Funnel. |

The bath or heated vessel consists of two flat-bottomed copper cylinders (24 B.W.G.), an inner one of 3 in. in diameter and $2\frac{1}{2}$ in. in height, and an outer one $5\frac{1}{2}$ in. in diameter and $5\frac{3}{4}$ in. in height; they are soldered to a circular copper plate (20 B.W.G.) perforated in the centre, which forms the top of the bath in such a manner as to enclose the space between

the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about $\frac{3}{8}$ in., that is, its diameter is about $\frac{3}{8}$ in. greater than that of the body of the bath, while the diameter of the circular opening in the centre is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, the screws being sunk below the surface of the ebonite to avoid metallic contact between the bath and the oil cup. The exact distance between the sides and bottom of the bath and of the oil lamp is $\frac{1}{2}$ in. A split socket, similar to that on the cover of the oil cup, but set at a right angle, allows a thermometer to be inserted into the space between the two cylinders. The bath is further provided with a funnel, an overflow pipe, and two loop handles. The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder or jacket (24 B.W.G.), flanged at the top, and of such dimensions that the bath while firmly resting on the iron ring just touches with its projecting top the inward turned flange. The diameter of this outer jacket is $6\frac{1}{2}$ in. One of the three legs of the stand serves as a support for the spirit lamp, attached to it by means of a small swing bracket. The distance of the wick-holder from the bottom of the bath is 1 in. Two thermometers are provided with the apparatus, the one for ascertaining the temperature of the bath, the other for determining the flashing point.

The thermometer for ascertaining the temperature of the ater has a long bulb, and a space at the top; its range is from about 90° to 190° F. The scale (in degrees of Fahrenheit) is marked on an ivory back, fastened to the tube in the usual way. It is fitted with a metal collar fitting the socket, and a part of the tube below the scale should have a length of about $3\frac{1}{2}$ in., measured from the lower end of the scale to the end of the bulb. The thermometer for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a round bulb, a space at the top, and ranges from about 50° to 150° F. It measures from end of ivory back to bulb $2\frac{1}{4}$ in.

Note.—A model apparatus is deposited at the Weights and Measures Department of the Board of Trade.

Directions for Applying the Flashing Test.—(1) The test apparatus is to be placed for use in a position where it is not exposed to currents of air or draught.

(2) The heating vessel or water bath is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be about 130° F., and this is attained in the first instance either by mixing hot and cold water in the bath, or in a vessel from which the bath is filled until the thermometer which is provided for testing the temperature of the water gives the proper indication; or by heating the water with the spirit lamp (which is attached to the stand of the apparatus) until the required temperature is indicated. If the water has been heated too highly, it is easily reduced to 130° F. by pouring in cold water little by little (to replace a portion of the warm water) until the thermometer gives the proper reading. When the test has been completed, this water-bath is again raised to 130° F. by placing the lamp underneath, and the result is readily obtained while the petroleum cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus, and the next test is proceeded with.

(3) The test lamp is prepared for use by fitting it with a piece of flat-plaited candle wick, and filling it with colza or rape oil to the lower edge of the opening of the spout or wick tube. The lamp is trimmed so that when lighted it gives a flame of about 0.15 of an inch in diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. When gas is available it may be conveniently used in place of the little oil lamp, and for this purpose a test flame arrangement for use with gas may be substituted for the lamp.

(4) The bath having been raised to the proper temperature,

the oil to be tested is introduced into the petroleum cup, being poured in slowly until the level of the liquid just reaches the point of the gauge which is fixed in the cup. In warm weather the temperature of the room in which the samples to be tested have been kept should be observed in the first instance, and if it exceeds 65° F. the samples to be tested should be cooled down (to about 60° F.) by immersing the bottles containing them in cold water, or by any other convenient method. The lid of the cup with the slide closed is then put on, and the cup is placed into the bath or heating vessel. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position is not under any circumstances to be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

(5) The test lamp is then placed in position upon the lid of the cup; the head line or pendulum, which has been fixed in a convenient position in front of the operator, is set in motion; and the rise of the thermometer in the petroleum cup is watched. When the temperature has reached about 60° F., the operation of testing is to be commenced, the test flame being applied once for every rise of 1° F. in the following manner:—

The slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation.

Note.—If it is desired to employ the test apparatus to determine the flashing points of oils of very low volatility, the mode of proceeding is to be modified as follows:—

The air chamber which surrounds the cup is filled with cold water to a depth of 1½ in., and the heating vessel or water bath is filled as usual, but also with cold water. The lamp is then placed under the apparatus and kept there during the entire operation. If a very heavy oil is being dealt with, the operation may be commenced with water previously heated to 120° F., instead of with cold water.

Fractional Distillation.—In addition to obtaining the specific gravity and flashing points of mineral burning oils, the

amount of distillates with their specific gravities on fractional distillation, would be a valuable means of judging their qualities. They should yield a comparatively small quantity of distillate below 150°C ., and a small percentage of heavy oils as a residuum at 300°C . A good method for the fractional distillation of burning oils is that recommended by Biel:—250 grms. of the oil are weighed into a 500 cc. flask which is covered with brass wire gauze to maintain an even temperature. It is connected to a Glinsky's dephlegmator and a Liebig's condenser. A thermometer is fixed in the dephlegmator, the bulb of which is to be on a level with the exit tube. A small flame is lighted under the flask, and the distillate up to 150°C . is collected in a weighed receiver and weighed. A second weighed receiver is replaced on the condensing tube and the flame enlarged, and the heating continued until practically all has distilled up to a temperature of 270°C ., which is weighed. The difference between the weights and the 250 grms. taken, may be considered the "tailings" or heavy oils left in the retort at 270°C .

Biel obtained the following results by the above process with different Russian burning oils:—

	Kerosene.			Pyronaphtha.	
	A.	B.	C.	D.	E.
Specific Gravity820	.820	.835	.857	.867
Flash Point $^{\circ}\text{C}$	52.5	35.0	44.5	7.5	94.0
Light Oil (below 150°) . .	0.8%	10.0%	6.0%	0.0%	0.0%
Normal Oils (150° to 270°) .	92.0%	76.5%	63.5%	44.5%	30.5%
Heavy Oils (tailings) . . .	7.2%	13.5%	30.5%	55.5%	69.5%

The following results were obtained by Biel with American and Russian burning oils:—

	American.		Baku.	
	No. 1.	No. 2.	No. 1.	No. 2.
Specific Gravity	·795	·783	·803	·822
Flash Point °C.	26	48	26	30
Light Oil (below 150° C.)	14·4 %	2·2 %	33·5 %	12·8 %
Normal Oils (150° to 270° C.) . .	45·9 %	87·8 %	66·5 %	78·3 %
Heavy Oils (tailings)	39·7 %	10·0 %	..	8·4 %

The following table by Redwood* shows the distinctive differences between Russian and American burning oils (kerosene). Nine fractions of 10 per cent. each were distilled off, and their respective specific gravities taken at 15° C., and also the 10 per cent. of residuum left in the retort :—

Original Sample.	Russian Kerosene. Specific gravity ·822.	American Kerosene. Specific gravity ·803.
1st fraction	·783	·748
2nd "	·796	·759
3rd "	·803	·778
4th "	·814	·792
5th "	·827	·802
6th "	·831	·812
7th "	·837	·822
8th "	·838	·831
9th "	·846	·838
10th " (residue)	·864	·849

In judging the quality of various burning oils for lighting railway carriages, &c., it would be wise to make a practical test under the same conditions as in actual use, noting :—

- (1) The intensity of light at start ;
- (2) The intensity of light towards the end ;
- (3) The consumption of oil per hour ;
- (4) The condition of wick at finish ;

comparing with what is considered a standard oil under the same conditions.

* *Four. Soc. Chem. Ind.*, iv., 76.

COMPARATIVE COST OF PETROLEUM AS AN ILLUMINANT.

The following table by Redwood, shows the comparative cost of a given amount of light from petroleum oil, colza oil, and coal gas, equal to that of a standard sperm candle, burning at the rate of 120 grains per hour for 1,000 hours :—

COST OF 1,000 CANDLE HOURS.

From Petroleum Oil.				From Colza Oil.			From Coal Gas.		
Price per gallon.				Price per gallon.			Price per 1,000 cubic ft.		
d. 9	s. d. 1 0	s. d. 1 3	s. d. 1 6	s. d. 3 0	s. d. 3 6	s. d. 4 0	s. d. 3 0	s. d. 3 6	s. d. 4 0
d. 7½	d. 9¾	d. 11½	s. d. 1 3	s. d. 2 4	s. d. 2 8	s. d. 3 0¾	s. d. 1 3	s. d. 1 5½	s. d. 1 8

From the above figures it will be seen that, taking petroleum at 1s. per gallon and coal gas at 3s. 6d. per 1,000 cubic feet, a given amount of light from the latter costs about twice as much as the former; and taking colza oil at 3s. 6d. per gallon and petroleum oil at 1s. per gallon, it will be seen that petroleum has the advantage threefold for a given amount of light.

Reporting upon the relative cost and efficiency of electricity, gas, and oil as illuminants, the Trinity House Committee conclude, that "for all practical purposes, gas and oil are equal," and that, "for the ordinary necessities of lighthouse illumination, mineral oil is the most suitable and economical illuminant."

THE STORAGE OF PETROLEUM.

Owing to the high degree of inflammability of petroleum oils, it is of importance that great care should be exercised in their storage. The following are some of the best means of safely storing petroleum :—

(1) The oil to be kept in metallic tanks, sunk some distance into the ground, from which quantities can be drawn as required with the aid of a hand pump.

(2) If the ordinary wooden barrels be desired to be kept intact, they should be kept in isolated warehouses of one storey only, built of unflammable materials.

(3) Weights are attached to the ordinary barrels, which are then sunk in water.

(4) A convenient mode of storing inflammable oils, is to place a vessel similar to a gasholder, mouth downwards into a large tank filled with water, in which the oil can thus be stored over the water, from which quantities can be drawn as required.

Notes on the Construction of Petroleum Lamps.—The Metropolitan Board of Works (now London County Council) have issued the following instructions as to the construction and management of ordinary mineral-oil lamps :—

(A) Lamps.

(1) That portion of the wick which is in the oil reservoir should be enclosed in a tube of thin sheet-metal, open at the bottom, or in a cylinder of fine wire gauze, such as is used in miners' safety-lamps (28 meshes to the inch).

(2) The oil reservoir should be of metal rather than china or glass.

(3) The oil reservoir should have no feeding-place or opening, other than the opening into which the upper part of the lamp is screwed.

(4) Every lamp should have a proper extinguishing apparatus.

(5) Every lamp should have a broad and heavy base.

(B) Wicks.

(1) Should be soft, and not tightly plaited.

(2) Should be dried at the fire before being put into lamps.

(3) Should be only just long enough to reach the bottom of the oil reservoir.

(4) Should be so wide that they quite fill the wick-holder without having to be squeezed into it.

(C) *Management.*

(1) The reservoir should be quite filled with oil every time before using the lamp.

(2) The lamp should be kept thoroughly clean. All oil should be carefully wiped off, and all charred wick and dirt be removed, before lighting.

(3) When the lamp is lit, the wick should be first turned down and then slowly raised.

(4) Lamps which have no extinguishing apparatus should be put out as follows:—The wick should be turned down until there is only a small flickering flame, and a sharp puff of breath should then be sent *across* the top of the chimney but not *down* it.

(5) Cans or bottles used for oil should be free from water and dirt, and should be kept thoroughly closed.

OILS FOR VARIOUS PURPOSES.

Gas Oils.

Oils obtained from crude petroleum and bituminous shale oil are now largely used in the manufacture of gas for the lighting of railway carriages, &c. The great point in the selection of oils for this purpose, is to ensure uniformity of supplies, when once the right kind of oil has been decided upon that is suited to the plant used.

The following is the specification of the Great Eastern Railway Company for shale gas oil, for use with their Pintsch apparatus:—

“The specific gravity to be not less than .855 at 60° F.

(= 15.5° C.). When subjected to fractional distillation, free ebullition is to commence at a temperature of about 550° F. (= 287.8° C.), and the first tenth by volume of the distillate is not to exceed .840 sp. gr. at 60° F. (= 15.5° C.). The succeeding tenths by volume of distillates are to exhibit a fairly uniform rate of increase in density, and the specific gravity of the tenth or final fraction, is not to exceed .880 at 60° F. (= 15.5° C.)."

The following results were obtained for three samples of shale gas oil supplied by three different firms, in response to inquiries for quotations for gas oil on the basis of the above specification:—

	No. 1.	No. 2.	No. 3.
Specific gravity at 15.5° C.869	.847	.887
Commencement of free ebullition . 287.8° C.	287.8° C.	287.8° C.	(above) 287.8° C.

Results of Distillations :—Specific gravity of fractions at 15.5° C.

No. of fraction.	No. 1.	No. 2.	No. 3.
1	.843	.830	.863
2	.845	.834	.868
3	.848	.837	.869
4	.859	.838	.875
5	.861	.841	.878
6	.866	.843	.880
7	.871	.847	.885
8	.878	.851	.889
9	.879	.856	.893
10	.880	.874	.906

The following are some interesting results obtained by Dr. Macadam on experimenting with Pintsch's and Keith's apparatus :—

BLUE PARAFFIN OIL USED.

	Pintsch's Apparatus.			Keith's Apparatus.		
	A	B	Average.	A	B	Average.
Specific gravity of oil . .	877.6	878.2	877.9	874.1	877.6	875.9
Flashing point	296°	294°	295°	292°	286°	289°
Firing point	356°	352°	354°	348°	346°	347°
Gas per gallon, cubic feet	90.7	103.4	97	85	84.8	84.9
Illuminating power . . .	62.5	59.1	60.8	63.2	59.5	61.4
Volume of oil flowing into each retort per hour	1.4	1.18	1.29	2.3	1.3	1.8
Gas per retort per hour .	126.8	122.5	124.6	197.5	111.9	154.7
Heavy hydrocarbons per cent.	39.2	37.1	38.2	39.9	38.2	39.0
Gas per ton (cubic feet) .	23,128	26,356	24,742	21,772	21,671	21,721

Oil of Turpentine ($C_{10}H_{16}$).

Pure oil of turpentine is obtained by distilling the oleo-resinous juice got from the bark, &c., of pine and fir trees, &c., and which yields from 10 to 30 per cent. of turps, leaving a residuum of resin or colophony.

American oil of turpentine has a specific gravity of .864 to .870, and commences to boil at about 155° C., and on distillation it completely passes over below 180° C.

Turpentine is often largely adulterated with petroleum products or "turpentine substitutes."

The writer recently examined a sample of supposed genuine turpentine, and found it to be wholly American kerosene. A good practical test of the drying properties of turpentine, is to drop a little of the sample on a piece of writing paper and allow it to evaporate at the ordinary temperature; genuine turpentine completely evaporates in a short time without leaving a trace of a greasy stain, while if kerosene, &c., be present, a more or less permanent greasy stain will be

produced which, on gently heating, will give an odour characteristic of its nature.

In making an assay of a sample of turpentine, the following are the most important points to be ascertained:—

(1) *The Specific Gravity*.—Genuine turpentine should have a specific gravity of $\cdot 864$ to $\cdot 870$; if it is much lower than this, it is almost sure to contain foreign hydrocarbons.

Resin spirit has a sp. gr. of	$\cdot 856$ to $\cdot 880$.
Shale naphtha . . .	$\cdot 700$ „ $\cdot 750$.
Coal tar solvent naphtha	$\cdot 860$ „ $\cdot 876$.
Petroleum naphtha . .	$\cdot 700$ „ $\cdot 745$.

(2) *The Flashing Point*.—American oil of turpentine flashes at about 34° C., and if any petroleum spirit or naphtha be present it would be considerably lower.

(3) *The Boiling Point*.—The range of the boiling point of genuine turps would be from 155° to 180° C.; while if petroleum products were present, the range would be considerably more.

(4) *Distillation*.—Distil 100 cc. of the sample in a 150 cc. retort, to 180° C., and collect the distillate in a graduated tube. With good turps at least 97 per cent. should come over at this temperature, and the residue in the retort would be oxidized products of the turpentine; if there is any considerable residue left in the retort at this temperature, it points to adulteration with hydrocarbon of higher boiling point. Obtain its specific gravity with a Sprengel tube. By distillation in a current of steam pure turps leave a small residue of about 5 per cent.; genuine old samples, however, leave as much as 2 per cent. residue, owing to the turpentine having absorbed atmospheric oxygen, forming resinous substances. If any petroleum is present it is unvolatilized, and may be recognised by its low specific gravity as compared with that of turpentine.

For the determination of petroleum in turpentine the following method* will be found to give fair results:—

A balloon flask of 750 cc. capacity is fitted with a two-hole

* Vide *Oil and Colourman's Journal*, Dec., 1890.

cork stopper. Through one hole is inserted the tube of a glass-stoppered drop funnel, having a capacity of 100 cc. The flask is also connected with an inverted condenser. About 300 cc. fuming nitric acid of sp. gr. 1·4 are placed in the flask, and 100 cc. of the turpentine to be tested are measured into the drop funnel. The flask is surrounded by cold water, and the turpentine is allowed to drop slowly into the nitric acid. As each drop strikes the acid violent action takes place, with evolution of red fumes. It is well to shake the flask occasionally during the operation. When the turpentine has all passed into the flask, the apparatus is allowed to stand until all action is over. The contents of the flask are transferred to a large separating funnel, and treated with successive portions of hot water. In this way all the products resulting from the action of the acid on the terpenes are removed, while any petroleum paraffins remain insoluble in water, and can readily be separated and measured.

Having procured some turpentine known to be free from petroleum, mixtures of the pure turpentine with samples of refined petroleum of various boiling points were prepared.

In order that judgment in experimenting might not be influenced by knowing the quantities of material used, the proportions employed in the mixtures were withheld from the operator.

Ten mixtures were analysed, with the following results :—

Pure turpen- tine used.	Petroleum used.	Boiling point of Petroleum.	Petroleum found by method described.
cc.	cc.	deg.	cc.
65	35	about 250	34·1
80	20	250	18·9
70	30	200	29
80	20	200	18·5
90	10	200	8·9
80	20	100	17·8
70	30	100	28·4
85	15	100	13·5
80	20	75	17·9
70	30	75	28

It will be noticed that the sum of the amounts of petroleum and turpentine used would be 100 cc. in each case, so that the number of cubic centimetres of petroleum found would also express the percentage of adulterant. It appears from the above results that the petroleum fractions, which boil at about 250° , are least affected by the fuming nitric acid, while the low-boiling fractions are affected the most.

But in order to keep the specific gravity of the turpentine where it should be, the higher boiling petroleum fractions must be used in the adulteration—a fact which is favourable for the application of this method in the analysis of such mixtures.

Rape Oil.

Rape oil or *Colza oil* is obtained from the seeds of various species of *Brassica*, chiefly those of *B. campestris* and *B. napus*. The principal applications of rape oil are for illuminating purposes, lubrication, and in the manufacture of india-rubber. It has a brownish-green or yellowish-brown colour, and a characteristic odour. The specific gravity of genuine rape oil ranges between .913 and .916 at 15.5° C.; if the density of a sample of rape oil be above .916 it is highly probable that it is adulterated with oils of higher density and less value, such as the drying oils, of cottonseed, linseed, sunflower, hempseed, &c., whose specific gravities range between .920 and .937. When rape oil is intended for lubrication, the presence of drying oils is highly objectionable, since they increase the gumming properties of the oil. A useful test for the drying properties of rape oil, is to expose a thin film of the oil on a glass plate at a temperature of 100° C. for twenty-four hours by the side of a sample of genuine rape, frequently noting the condition of the samples by touching them with the finger. Genuine rape remains comparatively fluid for a few days; but if any drying oil be present it will assume the consistency of a jelly, or in some cases become almost as hard as a resin. Another useful test for the drying properties of rape oil is the Elaïden test described on page 222, under "Various Tests."

The viscosity of the sample at 15.5° C. is another valuable point in judging of its quality, 50 cc. of genuine rape oil taking about 535 seconds to run out of Redwood's viscosimeter at 15.5° C. See table on page 188. Maumené's temperature reaction, described on page 222, would be useful in confirming the presence of some foreign seed oils, the average rise of temperature for genuine rape being about 60° C. by this test.

In addition to the above tests apply the following :—

(1) Ascertain the melting and congealing points of the total fatty acids, as directed on page 197. The fatty acids of good rape oil melt at about 20° C., and congeal at about 17° C.

(2) Determine the percentage of free fatty acids, as described on page 192. The proportion of free fatty acids in good rape oil varies from 0.5 to 4 per cent. Excess of free fatty acids is undesirable when the oil is intended for lubrication, owing to their action on bearings, &c., forming hard metal soaps; and when such an oil is used for illuminating purposes it burns with a smoky flame and chars the wick. If required for soap-making the presence of free fatty acids is less objectionable, since the acids are neutralized in the process.

(3) Test for free mineral acids. The presence of free mineral acids is very objectionable, if the oil is proposed to be used for lubrication. Traces of free sulphuric acid are often found in rape oils, owing to its incomplete elimination after refining.

(4.) Test for mineral oils as directed on page 194.

Olive Oil.

Olive oil, salad oil, or sweet oil is obtained from the fruit of the *Olea Europæa* by compression, or by extracting with bisulphide of carbon, in which it is soluble. It should be of a greenish-yellow colour, without odour, and of an agreeable taste, and non-drying. Its principal uses are for cooking, wool-dressing, lubricating, medicine, and soap manufacture. Its specific gravity ranges between .914 and .918. At a tem-

perature of 0° C. it congeals, which on compression yields one-third of its weight of solid fat (Palmitin and Stearin), melting at from 20° to 28° C.; the two-thirds of the fluid portion (olein), remain liquid at from -4° to -10° C. Olive oil, when long exposed, assumes an objectionable rancid odour, owing to the incomplete elimination of mucilaginous matters. In judging of the quality of a sample of olive oil, obtain the following points, as described under their respective heads, referring to the various tables to see how near to, or how far from, the genuine article it corresponds:—

- (1) Specific gravity at 15.5° C.
- (2) Temperature at which fat is deposited.
- (3) The percentage of free fatty acids (average should be 5 to 8 per cent).
- (4) Maumené's temperature reaction.
- (5) Test for mineral oils.
- (7) Test for fish oils.
- (8) The drying properties, Elaiden test.
- (9) Mineral acids.
- (10) The viscosity at 15.5° C.
- (11) The odour on heating.
- (12) Taste.
- (13) Acid tests.

Linseed Oil.

This well-known and useful oil is obtained from the seeds of the flax plant—*Linum usitatissimum*—principally grown in India and Russia. It is largely used for paints and varnishes, and in the manufacture of oil-cloth, rick-covers, and soft soap. It is used in four states, according to the purpose for which it is to be applied, viz.:—the raw, refined, artists', and the boiled oil. The colour of the raw oil is yellowish-brown, and its specific gravity varies from .930 to .937. At a temperature of -18° C. a little fat separates out, and it becomes perfectly congealed at -27° C. The most valuable characteristic of linseed oil is the facility with which it absorbs

atmospheric oxygen, with the formation of a tough, resinous-looking substance, materially differing in properties from the original oil. The drying properties of linseed oil are increased after it has been heated to about 180°C. , with a current of air passing through, and the heat continued until the oil is seemingly in a state of ebullition owing to the liberation of decomposition products. The addition of drying agents, such as sugar of lead, oxide of manganese, litharge, iron oxide, &c., during the heating, increases the drying properties of the finished or "boiled" oil, owing to their acting as carriers of oxygen.

In judging of the quality of a sample of linseed oil, its drying property is one of the most important points to be determined, and may be ascertained by smearing a glass plate with the sample, allowing it to be exposed in a water oven, and noting how long it takes to dry, and the nature of the residuum left as compared with that of a standard sample. If it be desired to ascertain if any driers have been added, and their nature, gradually ignite about 50 grms. of the sample in a large porcelain crucible, and make a qualitative analysis of the residue.

With regard to other tests of its quality, apply those given under the head of olive oil.

Cottonseed Oil.

This is a drying oil obtained from the *Gossypium barbadense*, and is largely used to adulterate rape oil, olive oil, and other oils. It is also used in cooking, soap-making, and in the manufacture of butterine. The specific gravity of cottonseed oil ranges from '922 to '930, and solidifies at from 1°C to 4°C. The melting point of the fatty acids from cottonseed oil is so high as 38°C. It appears that cottonseed oil is not likely to be adulterated itself, but a knowledge of the properties of the oil is of great importance, in ascertaining its presence in other oils. By applying the tests given under those of olive oil, cottonseed oil could be approximately estimated in a mixture.

VARIOUS TESTS FOR OILS.

Maumené's Temperature Reaction of Oils.—The principle of the test depends upon the fact that when 36 E sulphuric acid is mixed with a fatty oil, decomposition takes place, with considerable rise in the temperature, which varies with different oils. The following is the method of applying the test:—50 grms. of the oil are weighed into a 200 cc. tall beaker, and packed with cotton wool into a litre beaker. The temperature of the oil is next observed, and 10 cc. of 36 E H_2SO_4 , at the same temperature, gradually run in from a pipette or burette, which should occupy about 60 seconds to run out, the oil being thoroughly stirred with the thermometer the whole time; continue stirring after the addition of the acid, and note the highest temperature the thermometer indicates; on subtracting from this, the initial temperature, the increase in temperature will be obtained. The following are the results obtained by Maumené by the above process with various oils:—

	Rise in Temperature °C.
Olive oil	42
Almond oil	52-54
Rape „	57-58
Arachis „	67
Beechnut oil	65
Sesamé „	68
Poppyseed oil	74
Hempseed „	98
Walnut „	101
Linseed „	103
Castor „	47
Tallow „	41-44
Horsefoot „	51
Cod liver „	102-103
Skate liver „	102

Poutet's Elaidin Test.—This is a very useful test for detecting drying oils mixed with non-drying oils and *vice versa*.

It depends upon the formation of elaidin (an isomeric modification of olein, and which is solid at the ordinary temperature), when a non-drying oil is subjected to the action of nitrous acid, the drying oils, which consist largely of linolein, being not so affected. The following is the manner in which the test is performed :—Measure out 50 cc. of the oil into a wide-mouthed stoppered bottle of about 100 cc. capacity, and pour on to the oil 2 cc. of a cold freshly-made solution of nitrate of mercury, prepared by dissolving 13 grms. of mercury in 12 cc. of 16 E nitric acid, the beaker in which it is dissolved being kept in cold water to retain nitrous fumes. Agitate the mixture, and allow to stand in an air bath, regulated to 25° C., for twenty-four hours, with occasional shaking, together with a pure standard sample of a similar oil. Note the length of time that solidification takes place, also the consistency of the mass after standing for the twenty-four hours. The drying oils, such as linseed, hempseed, &c., remain fluid, while the non-drying oils, such as olive, tallow oil, almond, sperm, yield hard products ; and oils of the intermediate class, such as rape, neats-foot, sperm, cottonseed, whale, &c., leave a mass more or less of the consistency of treacle.

Sulphuric Acid Test.—This is the best of the various colour tests for oils recommended by Chateau. There are various methods of applying it, the most suitable being that recommended by Allen,* who adds one or two drops of 36 E H_2SO_4 in the centre of twenty drops of the oil, and notes the colour produced before and after stirring, when the following colours are produced with various oils :—

* *Vide* "Comcl. Org. Analysis." V. ii., p. 59.

Oil.	One or two drops of 36 E H_2SO_4 to twenty of the oil.	
	Before Stirring.	After Stirring.
<i>Vegetable Oils.</i>		
Olive oil	Yellow, green, or pale brown.	Light brown or olive-green.
Almond oil	Colourless or yellow.	Dark yellow, olive, or brown.
Earthnut oil	Greyish yellow to orange.	Greenish or reddish brown.
Rape oil (crude). .	Green with brown rings.	Bright green, turning brownish.
„ „ (refined) . .	Yellow with red or brown rings.	Brown.
Mustard oil	Dark yellow with orange streaks.	Reddish brown.
Cottonseed oil (crude)	Very bright red.	Dark red, nearly black.
„ „ (refined)	Reddish brown.	Dark reddish-brown.
Nigerseed oil	Yellow with brown clot.	Reddish or greenish-brown.
Poppyseed oil . . .	Yellow spot with orange streaks.	Olive or reddish-brown.
Linseed oil (raw) .	Hard brown or greenish-brown clot.	Mottled dark brown.
„ „ (boiled)	Hard brown clot.	Mottled dark brown.
Castor oil	Yellow to pale brown.	Nearly colourless or pale brown.
<i>Animal Oils.</i>		
Lard oil	Greenish-yellow or brownish with brown streaks.	Mottled or dirty brown.
Tallow oil	Yellow spot with pink streaks.	Orange red.
Whale oil	Red, turning violet.	Brownish-red, turning brown or black.
Seal oil	Orange spot with purple streaks.	Brownish-red, changing to mottled brown.
Cod liver oil	Dark red spot with purple streaks.	Purple, changing to dark brown.
Sperm oil	Pure brown spot with faint yellow ring.	Purple, changing to reddish or dark brown.
<i>Hydrocarbon Oils.</i>		
Petroleum lubricating oil.	Brown.	Dark brown, with blue fluorescence.
Shale lubricating oil	Dark reddish brown.	Reddish brown, with blue fluorescence.
Resin oil (brown) .	Bright mahogany brown.	Dark brown, with purple fluorescence.
„ „ (pale) . .	Mahogany brown.	Red brown, with purple fluorescence.

Massie's Nitric Acid Colour Test.—Mix 3 cc. of the oil for two minutes with 1 cc. of pure 16 E nitric acid, and observe the colour of the separated oil. The following are the colours produced with various oils by this method :—

Olive oil	Colourless, yellow or greenish.
Almond oil	Colourless or slightly greenish.
Arachis „	Reddish.
Peach-kernel oil . .	Immediate red liniment.
Rape oil	Reddish or orange.
Sesamé oil	Yellowish or orange.
Cottonseed oil . . .	Brown or brownish-red.
Linseed „	Red or orange.
Poppyseed „	Reddish.
Hempseed „	Brownish-red.
Castor „	Yellowish or orange.

Tests for Fish Oils in Vegetable Oils.—Heat some of the sample in a small covered crucible to about 130° C., and observe the odour given off. If much fish oil be present a characteristic fishy odour will be perceived. Measure out 50 cc. of the sample into a large test tube, and pass a current of dry chlorine gas through it, when a more or less reddish-brown colour will be produced if fish oils be present. Saponify 5 grms. of the sample as directed on page 194, but using absolute alcohol instead of the methylated, (which gives a brownish colour with potash, owing to the methyl alcohol it contains), and dilute the saponified oil to about 50 cc. with water in a test tube, when, if fish oils be present, a more or less brownish colour is produced.

Valenta's Acetic Acid Test.—In this test the oil is brought into solution by boiling in 17 E acetic acid, and the temperature at which a turbidity is produced is observed. This being different for various oils, the test may be useful as confirming the presence of a particular oil which has been previously identified by other means. The test may be applied in the following manner :—5 cc. of the sample and 5 cc. of glacial (17 E) acetic acid are poured into a test tube, and the

mixture heated to boiling, until all the oil has gone into solution; a thermometer is then immersed in the liquid, which is allowed to cool gradually, and the temperature at which a turbidity is produced is observed. The following results were obtained by Valenta, for various oils, by this test:—

Kind of Oil.	Temperature of Turbidity ° C.
Green Olive oil	85
Yellow „	111
Almond oil	110
Arachis „	112
Apricot Kernel oil	114
Sesamé oil	107
Melonseed oil	108
Cottonseed oil	110
Cod-liver „	101
Rape seed oil	Not completely dissolved at the boiling point of acetic acid.
Mustard seed oil	
Wild Radish-seed Oil	
Palm oil	23
Laurel „	26 to 27
Nutmeg Butter	27
Cocoa Nut oil	40
Palmnut oil,	48
Bassia oil	64.5
Beef Tallow	95
Pressed Tallow	114
Cacao Butter	105
Olive Kernel oil	Soluble at the ordinary temperature.
Castor oil	

Iodine Absorption Test for Oils.—This process of testing oils, which was first brought out by Baron Hübl* depends upon the fact that different oils, under the same conditions combine with varying quantities of iodine, owing to each oil possessing the glycerine ethers of the members of the three groups of fatty acids—viz., the *acetic*, *acrylic*, and *tetrolic* series, in different proportions; the acetic series being comparatively indifferent to the action of iodine, while the fats of the acrylic

* *Ding. Polyt. Jour.*, ccliii., 281.

and tetrolic series readily unite with fixed quantities of the halogen.

The following are details of the process :—

Weigh out into a 30 cc. beaker from 0.2 to 0.3 gm. of a drying oil, 0.3 to 0.4 gm. of a non-drying oil, or from 0.8 to 1.0 gm. of a solid fat. Dissolve in 10 cc. of chloroform, and pour into a 250 cc. stoppered flask. 20 cc. of a *standard* solution of iodo-mercuric chloride (prepared by dissolving 25 grms. of iodine and 30 grms. of mercuric chloride each in 500 cc. of 95 per cent. alcohol, and mixing the two solutions) are now added and the mixture well agitated. If the solution is not quite clear add a little more chloroform. If the mixture becomes nearly colourless after standing fifteen minutes add 10 cc. more of the iodine solution, well mix, and allow to stand for two hours. Now add 10 cc. of an aqueous solution of E potassic iodide, mix, dilute to about 200 cc., and titrate the

free iodine still present with an $\frac{E}{10}$ standard solution of hyposulphite of soda (thiosulphate, $Na_2S_2O_3$), using 2 cc. of starch solution (1 in 100) as indicator of the end of the reaction.

A blank experiment must be made, using the same quantities of chloroform, iodine solution, &c., in order to obtain the true value of the hyposulphite solution, after which the amount of iodine absorbed by the sample is easily obtained, which should be expressed centesimally.

The following table shows the iodine absorption for various oils in percentages (Hübl and Möller):—

							Iodine degree.
Shark-liver oil	268.2
Manhaden „	170.8
Porpoise „	131.2
Seal „	103.4
Linseed „	158
Walnut „	143
Poppyseed „	136
Cottonseed „	106
Rapeseed „	100
Almond „	98.4

	Iodine degree.
Castor oil	84·4
Olive „	82·8
„ „	81·3
Lard „	59
Palm „	51·5
Tallow „	40
Cocoa-nut oil	8·9
Cocoa-butter oil	34·4
Muskat-butter „	31·6
Mutton-fat oil	57·3
Lard „	55
Beef-suet „	38·4
Oleic acid „	86·2
Olein	82·3
Commercial stearin	1·7
Beeswax	5·3
Japanese wax	5·61
Bayberry tallow	1·38
Butter	31
Oleomargarine	55·3

Hübl obtained an assimilation of from 89·8 to 90·5 per cent. of iodine by chemically pure oleic acid, while the theoretical proportion required by the reaction $C_{18}H_{34}O_2 + I_2 = C_{18}H_{34}I_2O_2$ is 90·07 per cent.

The following are his conclusions deduced from the experiments* :—

(1) Linseed oil is distinguished by its high iodine, and any addition of a foreign oil must reduce that degree. By boiling linseed oil, its iodine degree is lowered, but the fusing point of its fatty acids is increased.

(2) An addition of 5 to 10 per cent. of cottonseed or rapeseed oil would be readily detected in nut or poppy oil, but not much less than 20 per cent. of linseed would be detected.

(3) Rapeseed oil as regards the iodine absorption is affected by the method of extraction and refining. The refined oil usually has an iodine degree 2 or 3 degrees below that of the

* See "Journal Soc. Chem. Ind.," 1884, p. 642.

raw product. A falsification with 15 per cent. of linseed oil would be detected with certainty.

(4) Castor oil possesses a very constant iodine degree — from 84.0 to 84.7, and is distinguished from all other oils and fats by this figure, by the points of fusion and solidification of its fatty acids (13° and 3° C. respectively), by the saponification test and by its ready solubility in alcohol and acetic acid.

(5) In twenty samples of olive oil, collected from various sources, the absorption of iodine varied only within 3 degrees. The addition of about 5 per cent. of a drying oil, or 15 per cent. of cottonseed, sesamé, arachis, or rape-seed, may be established with certainty.

(6) Animal fats, as is well known, are liable to alteration in respect to their consistency and proportion of oleic acid, owing to variety, age, and food of the animal whence derived. In the case of butter-fat the absorption of iodine varies between the limits of 26 and 35.

(7) When the nature of two fats in a mixture is known their proportion may be determined approximately by the formula :—

$$x = \frac{100(I-n)}{m-n}$$

Where x = the percentage of one fat.

y = " " the other.

I = iodine degree of the mixture.

m = " " fat x .

n = " " " y .

(8) The age of a fat, so long as great alterations have not taken place, does not affect its iodine absorption. If, however, an oil has become rancid and contains free acid, the iodine degree suffers considerable depression.

PART VI.

MATERIALS USED IN THE MANUFACTURE OF RAILWAY GREASE.

RAILWAY grease may be made from tallow, palm oil, petroleum residuum, caustic or carbonated alkali, and soap. A known weight of alkali (which is determined according to the percentage of free fatty acids present in the tallow and palm oil), is first dissolved in an appropriate quantity of water contained in an open boiler, into which is fitted a perforated coil of iron tubing, through which steam can be made to issue. A weighed quantity of palm oil is next added, and thoroughly stirred until the free fatty acids are completely neutralized, which can be known by the abatement of effervescence, due to CO_2 from the decomposition of the alkali; a definite weight of tallow is next added, and the mixture "boiled" up with steam for some time, when the free fatty acids of the tallow thus become neutralized. After the addition of soap, and steaming, the grease is run off through sieves into vats, or, if a black grease is required, about 40 per cent. of Russian petroleum residuum is previously added.

The quality of the materials used in the manufacture of grease is of great importance in ensuring uniformity of working, and in obtaining a homogeneous product. A source of trouble in practical working is the great difference in the percentages of free fatty acids in different consignments of palm oil, Lagos brand containing about 12 per cent. of free fatty acids,

expressed as palmitic acid, while the brand known as Salt Pond may contain as much as 80 per cent. of free fatty acids.

The following may be taken as being the average composition of good yellow and black railway grease:—

	Yellow. Per cent.	Black. Per cent.
Water	37	25
Tallow	30	17
Palm oil.	10·5	4
Soap (containing 30 % water)	21	13·5
Petroleum residuum . . .	—	40·0
Soda ash	1·5	1·5
	<hr/> 100·0	<hr/> 100·0

Grease used on the German railways has the following composition:—

	Per cent.
Tallow	24·6
Palm oil	9·8
Rape-seed oil	1·1
Soda	5·2
Water	59·3
	<hr/> 100·0

Austrian Grease:—

	Tallow.	Olive Oil.	Old Grease.
For winter	100	20	13
For spring and autumn . .	100	10	10
For summer	100	1	10

Tallow.

Australian mutton tallow and Russian tallow are much used in the manufacture of grease, and in judging its quality it becomes necessary to know the amount of moisture, free fatty acids, suspended matter (mineral and organic), the total fatty acids, together with the determination of the melting and congealing points of the sample, and also of the total fatty acids.

Estimation of Moisture.—Weigh into a tared beaker, supplied with a glass rod, 10 grms. of the fairly averaged

sample, dry in an oil bath or air oven for one hour, at a temperature of 110°C ., allow to cool and reweigh; replace in the oil bath or air oven for another hour, cool, and again weigh; if there is no considerable difference between this and the last weighing the loss is taken as moisture. The normal amount of moisture in tallow ranges from 0.5 to 2 per cent.

Estimation of Suspended Matter.—If the amount of suspended matter is at all considerable—which could be judged when the tallow is melted—the tallow left from the estimation of moisture is melted, and passed through a tared ashless filter-paper contained in a hot water funnel, after which it is transferred to an ordinary filter stand, and the remaining portion of tallow and suspended matter in the beaker treated with methylated ether, and filtered, the filter being washed free from fat with ether, dried and weighed; after which the filter and its contents can be ignited in a weighed crucible, and the residual mineral matter weighed. The total suspended matter in a tallow should not exceed 0.3 per cent.

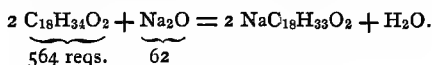
Estimation of Free Fatty Acids—50 grms. of the sample are weighed into a 180 cc. flask, and 100 cc. of pure methylated alcohol (to which are previously added three drops of an alcoholic solution of phenolphthalein and two drops of E NaHO solution, which should give a permanent pink coloration) are added; the mixture is then placed on top of a water oven, and when the tallow is seen to be melted, it is vigorously stirred so as to dissolve all the free fatty acids. A standard solution of E sodic hydrate, contained in a burette, is now gradually run into the solution until a pink coloration (which remains permanent on stirring for five minutes) is produced. Note the volume required. $1\text{ cc. of E NaHO} = 0.282\text{ gm. free oleic acid}$; cc.'s required $\times .282 \times 2 =$ percentage of free oleic acid. The usual range of free fatty acids in tallow is from 1 to 5 per cent., and in samples which have been kept for some years, and which are characterised with a very offensive rancid odour, may contain as much as 20 per cent. Sometimes, however, the free fatty acids are abnormally high, due to

adulteration with cotton-seed oil, stearic acid, and the acids from wool grease.

Estimation of Total Fatty Acids—5 grms. of the sample are saponified as directed on page 194, and the total fatty acids determined as described.

The Melting Point and Congealing Point of the Sample, and of the Total Fatty Acids therefrom.—These are determined by the method described on page 197. The melting point of Australian tallow is from about 45° to 50° C., and the congealing point from 40° to 47° C. The melting point of the total fatty acids is often 49° C., and the congealing point 47° C.

Calculation of amount of Soda-ash required to neutralize the Free Fatty Acids in 1 cwt. of Tallow.—We shall suppose the soda-ash to contain 47.7 per cent. of combined Na_2O and the tallow to contain 4.8 per cent. free oleic acid. Now two molecules of oleic acid require one molecule of sodic oxide, since



Now 1 cwt of the tallow contains .048 cwt. $\text{C}_{18}\text{H}_{34}\text{O}_2$; then $564 : .048 :: 62 : x = .00528$ cwt. pure Na_2O required, but the soda-ash only contains 47.7 per cent. pure Na_2O ; then, $47.7 : 100 :: .00528 : x = .01107$ cwt. or $.01107 \times 112 = 1.24$ lbs. soda-ash required to neutralize the free fatty acids in 1 cwt. of the tallow.

Palm Oil.

Palm oil is obtained from the fruit of the *Avoira elais* or *Elais Guineensis*, and is imported largely into this country from the West Coast of Africa for the purpose of making grease, soaps and candles, &c. Its specific gravity is .920 to .927 at 15° C. and about .856 at 100° C., and it melts at about 40° C., the total fatty acids melting at from 41 to 46° C.

The colour of palm oil varies from orange yellow to brownish yellow. In consistency it varies from that of vaseline to hard tallow, owing to the variation in the proportion of free

fatty acids. Palm oil may be assayed in a similar manner to tallow, the free fatty acids being expressed as palmitic acid, ($C_{16}H_{32}O_2$) 5 grms. instead of 50 being taken. For railway grease the amount of free fatty acids present is not a serious item, since they are neutralized in the process of manufacture. The oil in its raw state, however, quickly corrodes iron, steel, &c. The following is a table by Archbutt giving the proportions of free fatty acids in various brands of palm oil.

	Free Fatty Acids, per cent. Expressed as Palmitic.
Salt Pond	78.9
Unknown	72.0
Refined	55.8
Brass	53.2
New Calabar	52.2
Fernando Po	40.5
Half Jack	35.7
Half Jack	24.4
Bonny	21.5
Lagos	11.9

The following is what the author considers an ideal palm oil for grease making :—

Free fatty acids	30 per cent.
Total „ „	95 „
Water, below	0.5 „
Suspended matter, below3 „
Melting point of sample	41° C.
Congealing point of sample	36° C.
Melting point of total fatty acids	42° to 47° C.
Congealing „ „	43° to 41° C.

The Lagos brand contains the least proportion of free fatty acids, but it is likewise the most expensive, and therefore it is more economic to use for grease making a palm-oil containing a fair proportion of free fatty acid at a reasonable price. It could, of course, be arranged that there should always be the same proportion of neutral palm fat present in the grease, by altering the proportion of added soap according to the amount

of soap formed by the neutralization of the free fatty acids. There should be no difficulty in the making of grease with palm oil so long as the proportion of the free fatty acids keeps below 40 per cent.

Petroleum Residuum.

The best petroleum residuum applicable to grease-making is that imported from Russia. If the distillation has not been carried too far, it is of a fine glossy silky nature ; and when rubbed on the hand, it exhibits a good greasy consistency. On the other hand, if the distillation has been carried on to the extent that the last portion of lubricating oil has been removed, a rough-feeling product is obtained which is inferior for the purpose.

Petroleum residuum is now much used in the manufacture of railway grease, and it appears to be a very economic introduction. It can be got for something like £6 per ton ; and moreover, the grease made with it lasts longer, and has less action on the bearings, journals, &c., than the ordinary yellow grease. The most important point in buying residuum is to get it of the required viscosity ; many of the residuums in the market become very thin when the temperature rises a little, and grease made with them is of little value, since on attaining the temperature of the bearings it separates and runs away in waste. The viscosity of residuums for grease-making should be determined at 180° C., and the best for the purpose takes about 546 seconds for 50 cc. to run out of Redwood's viscosimeter. The amount of suspended matter if large must be determined, and also the loss on exposing about 1 grm. of the sample contained in a watch glass of 2½ inches diameter, in a water oven for twelve hours, the loss being due to water and any light oil that has volatilized, an amount which rarely exceeds 2 per cent. Sometimes residuums contain a considerable amount of water ; in such cases it is best to treat a known weight with petroleum ether in a graduated measure, and note the volume of water that has separated out.

Soap.

Palm soap containing 28 per cent. of water and 8 to 13 per cent. of resin as soap is much used for grease-making, but it is questionable whether a much cheaper soap could not be used with equal advantage, containing no resin, which is of little use as a lubricant.

METHOD OF ANALYSIS.

Estimation of Water.—The amount of water in soaps considerably varies, some hard soaps containing 15 per cent., and inferior varieties as much as 80 per cent. Weigh out 2 grms. of the fairly averaged sample into a tared beaker supplied with a pointed glass rod, and dry for one hour at 50° C.; and finally gradually increase the temperature to 110° C., with frequent stirring until all water has been driven off; about three hours in all being in most cases sufficient to completely expel water. The loss = water.

Estimation of Total Combined Fatty and Resin Acids.—5 grms. of the average sample are dissolved in 100 cc. of hot water and poured into a separator, 20 cc. of standard E HNO_3 are then added, and the mixture well shaken up; the soap is thus decomposed, fatty acids and any free unsaponified fat being liberated, the soda or potash combining with the nitric acid forming nitrate of soda or potash. 50 cc. of methylated ether are now added, well shaken, and allowed to settle. The aqueous solution is tapped off into another separator, 20 cc. more ether added to it and separated as before; the two ethereal solutions are mixed and washed with water, and separated, adding the aqueous solution to the main portion. Reserve aqueous solution. The ethereal solution containing the fatty acids, &c., is poured into a weighed flask and attached to a Liebig's condenser, the ether distilled off, and the residue carefully dried and weighed; any unsaponified fat and resin as found in another portion, must be subtracted from this weight. Of course the fatty acids as thus found do not exist in this

form in the soap, but as fatty anhydrides; to obtain the corresponding quantity of fatty anhydrides; multiply the weight of fatty acids found by .966.

*Estimation of Total Soda (Na_2O).—*To obtain the total amount of soda present, free and combined, the reserved aqueous solution from the above determination is taken: the excess of E nitric acid therein is titrated back with a standard E solution of NaHO , using litmus paper as an indicator.

Each cc. of E HNO_3 neutralized = .032 grm. Na_2O .

The solution could now be divided into two halves, and if necessary the chlorides could be estimated in one by precipitation with nitrate of silver, and sulphates in the other by precipitation with barium chloride in the usual manner.

Estimation of Resin.—The best method for the determination of resin is that devised by Gladding, depending upon the solubility of silver resinate in an alcohol-ether solution, and the comparative insolubility of silver oleate, stearate, palmitate, &c., in the same solution. Weigh out 0.5 grm. of the total fatty and resin acids, as obtained in their determination, into a small flask, dissolve in 20 cc. of absolute alcohol, add two or three drops of phenolphthalein, then add drop by drop a concentrated alcoholic solution of potash till a pink coloration is produced, and then add two drops more, and keep near the boiling point for ten minutes to ensure complete saponification of any neutral fat; pour out into a graduated 100 cc. Muter tube, Fig. 29, dilute to 100 cc. with anhydrous ether, and thoroughly mix. Now add 0.5 grm. of pure silver nitrate in the state of the finest powder, and thoroughly shake for about ten minutes to ensure complete reaction. Allow the precipitated silver soaps to subside. About 70 cc. or any known quantity of clear ethereal solution are tapped off into a separator, and shaken up with about

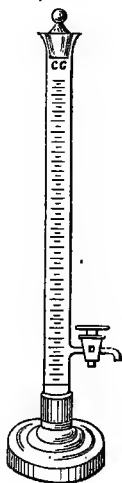


FIG. 29.

0.1 gm. more powdered silver nitrate to ensure complete formation of silver soaps. If any flocculent precipitate is produced the solution is again run into the Muter tube and shaken up with 0.3 gm. more of the powdered silver nitrate, allowed to settle, and a known volume tapped off into a separator as before. 20 cc. of 4 E HCl are now added to the liquid, and the mixture vigorously shaken up. The soluble silver soaps are thus decomposed, silver chloride is precipitated and collects at the bottom of the aqueous solution, while the resin acids and small quantities of oleic acid, &c., are dissolved in the ethereal layer. When complete separation is ensured, the aqueous solution with the precipitate is tapped off, the ethereal solution distilled off in a weighed flask, and the residue of resin, &c., dried at 100° C. and accurately weighed. Now, since the silver soaps of oleic, stearic acids, &c., are to an appreciable extent soluble in the ethereal liquor, a correction must be made for the solubility; the amount dissolved varies with the nature of the fatty acids.

The following table showing the average variation of solubility of different fatty acids as silver soaps, was obtained by Wright and Thompson :—

	Milligrams of Fatty Acids in 10 cc. of Solvent.
Pure oleic acid	1.20
Pure stearic acid	1.16
Mixed stearic and oleic acids	1.91
Acids from cottonseed oil	2.69
Acids from castor oil	5.39
Acids from cocoa-nut oil	1.80
Acids from linseed oil (Allen)	2.31

An idea of the nature of the fatty acids present having been obtained from their melting point, specific gravity, &c., the operator could judge which was the most correct figure to take for subtraction. The method gives good results with a little practice, but unless great care is exercised a very erroneous result may be obtained.

Estimation of Free Fat.—Owing to incomplete saponification of the fat used in the manufacture of soap, there is sometimes fat present in the free state. To determine this, the dry residue left from the estimation of water is detached by means of a flexible steel spatula, powdered, brushed into a 50 cc. wide mouth stoppered bottle, 30 cc. of methylated ether added, and the stopper tied down. It is now heated to about 40° C., well shaken for about a quarter of an hour, and allowed to cool; the stopper is then withdrawn, the clear ethereal solution decanted through a filter into a separator, the residue exhausted twice more with 30 cc. of ether, and filtered as before. The ethereal solution containing any free fat, free fatty acids, a little soap, &c., is now washed three times with 20 cc. of distilled water, the purified ethereal extract distilled, and the residuum of free fat, &c., weighed and tested for mineral oils and waxes, &c.

The following is a tabular scheme given by Dr. Leeds* for the complete analysis of soaps :—

* *Chem. News*, xlviii., 67.

Weight out 5 grms. of sample. Dry at 110° C. Loss = water. Treat with petroleum ether.

Residue is soap and mineral constituents. Treat with alcohol.

Residue. — Na_2CO_3 , NaCl , Na_2SO_4 , sodium silicate, starch and insoluble residue. Wash with 60 cc. water.

Filtrate. — Na_2CO_3 , NaCl , Na_2SO_4 , and sodium silicate. Divide into four equal parts.

Na_2CO_3 . Titrate with standard H_2SO_4 , and calculate as Na_2CO_3 .	NaCl . Titrate with standard AgNO_3 , or weigh as AgCl . Calculate as NaCl .	Na_2SO_4 . Precipitate with BaCl_2 , weigh the BaSO_4 , and calculate as for Na_2SO_4 .	Sodium silicate. Decompose with HCl , and determine combined mine soda to silicate and silica.
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Residue. — Starch and insoluble residue. Dry the filter and weigh; the weight is the starch and insoluble residue starch. Convert into $\text{C}_6\text{H}_{12}\text{O}_6$, titrate with Fehling's solution, subtract the weight of starch found, and the difference is the insoluble mineral constituents.

Extract is uncombined fat. Dry at 110° C., and weigh.

$\text{E H}_2\text{SO}_4$ used corresponds to free caustic alkali. Calculate as NaHO .

Filtrate. Combined soda and glycerine. Titrate excess of acid back with standard E NaHO .

After titration evaporate to dryness on the water bath. Treat with absolute alcohol, evaporate the alcoholic solution in a tared flask, and weigh any glycerine.

Precipitate is oleate, palmitate and stearate of silver. *Solution*. Resinate of Ag . Filter 50 cc. from total 100 cc., decompose with 20 cc. of E HCl . Allow to settle. Evaporate an aliquot part in tared beaker, dry at 110° C., and weigh. After applying the correction the weight = resin; subtract this from fatty acids + resin present; remainder = fatty acids.

SYNOPSIS OF RESULTS.

Per cent.

Fatty anhydrides
Resin acids
Soda, existing as soap
Water
Sodic carboate
hydrate
chloride
sulphate
Silica
Silica as silicate
Lime, &c.

* Equals per cent. fatty acids.

TABLE OF ANALYSIS OF VARIOUS SOAPS. (HOPE).*

Description of Soap.	Origin.	Fatty and Resin Anhydrides.	Soda Naso existing as Soap.	% Silica.	Soda as Silicate.	Caustic and Carbonate of Soda.	Sodic Chloride.	Sodic Sulphate.	Lime, Oxide of Iron, &c.	Water.	Total.	Fatty and Resin Acids.
White, No. 1	Tallow	69.06	8.98	0.01	nil	0.27	0.49	0.16	0.07	21.14	100.18	71.20
" " 2	Tallow and Coconut Oil	60.50	6.82	0.06	"	0.06	0.11	0.12	0.16	32.20	100.03	62.36
" " 3	Do.	55.71	6.90	0.03	"	0.92	0.18	trace	0.08	36.54	100.36	57.44
" " 4	Do.	44.27	6.23	7.02	2.36	0.75	0.32	0.34	0.34	38.14	99.77	45.64
Cold Water, No. 1	Tallow, Resin, and Cottonseed Oil	71.30	7.98	1.07	0.48	0.75	0.36	0.30	0.16	17.44	99.84	73.50
" " 2	Do.	49.95	7.00	2.34	1.01	0.33	0.51	nil	0.50	38.18	99.82	51.50
Olive Oil, No. 1	Olive Oil	71.20	7.58	0.06	0.03	0.22	0.66	0.17	0.20	19.70	99.82	73.40
Marseilles, No. 1	Chiefly Olive Oil	62.66	7.27	0.06	0.03	0.77	0.76	0.30	0.16	28.20	100.21	64.60
Palm Oil, No. 1	Palm Oil	59.28	6.65	0.42	0.01	0.39	0.47	0.13	0.16	32.35	99.86	61.08
Mottled	Palmnut Oil	38.89	5.76	6.40	1.29	1.62	1.78	0.72	0.03	38.70	95.19	40.10
Satinet.	Tallow and Resin	59.92	6.76	0.02	nil	0.92	0.41	0.37	0.05	31.30	99.75	61.77
Glasgow Almond.	Do.	42.41	4.14	5.64	1.59	2.76	0.37	trace	0.14	42.88	99.93	43.72
Pale Resin, No. 1	Do.	60.90	7.22	0.04	nil	0.10	0.46	0.12	0.02	31.22	100.08	62.78
" " 2	Do.	48.20	5.00	0.42	0.18	0.15	0.65	0.10	0.10	45.00	99.80	49.65
" " 3	Do.	39.92	4.70	0.62	0.25	0.20	1.48	0.18	0.15	52.40	99.90	41.15
Milling	—	63.06	7.25	0.02	nil	0.10	1.65	0.15	0.30	27.47	100.00	64.95
Yellow (for foreign markets)	—	10.90	1.36	0.03	"	trace	2.57	0.56	0.14	84.00	99.56	11.20
Marine (for emigrants)	Palmnut Oil	19.42	3.11	9.00	3.98	3.00	5.13	0.35	0.16	53.32	97.47	20.02

* Allen, "Comel. Organic Analysis," vii. p. 272.

Assay of Caustic Soda (NaHO).

Weigh out quickly 10 grms. of the fairly averaged sample, dissolve in the purest distilled water free from CO_2 , dilute to 1,000 cc. at 15°C . in a graduated stoppered flask, thoroughly mix, and allow any sediment to subside.

Estimation of Total Alkali.—100 cc. (= 1 gm. sample) of the above clear solution are measured off and poured into a 200 cc. beaker; 28 cc. of *standard E* H_2SO_4 are now run in from a burette, and the mixture boiled for five minutes with the cover on. It is then cooled, a few drops of litmus solution added; and *standard E* NaHO run in gradually from a burette, stirring until the last drop added just turns the red solution blue. Note the volume required, subtract this amount from 28, and the quantity of *E* acid neutralized by 1 gm. of sample is obtained.

Each cc. neutralized = $\cdot 031$ gm. soda (Na_2O).

Example.—A sample of caustic soda treated as above required 8.8 cc. of *E* NaHO to neutralize excess of acid, then:—

$$28 - 8.8 = 19.2 \text{ cc. E } \text{H}_2\text{SO}_4 \text{ neutralized.}$$

$$19.2 \times 0.031 = .5952 \text{ gm. Na}_2\text{O in 1 gm.}$$

$$.5952 \times 100 = 59.52 \text{ per cent. Total Na}_2\text{O.}$$

Estimation of Na_2O present as Hydrate and Carbonate.

—Take 250 cc. of the original solution (= 2.5 grms. of sample), pour into a graduated 500 cc. flask, add 40 cc. of *E* BaCl_2 , dilute to 500 cc. at 15°C ., stopper, thoroughly mix, and allow any precipitate of carbonate of barium to subside; decant off 250 cc. (= 1.25 gm. original sample) of the clear solution into a 400 cc. beaker, add three drops of phenolphthalein solution, and run in *standard E* HCl from a burette until the pink colour has just disappeared. Note the amount required.

Example.—250 cc. of the original solution were treated as above, and 22.6 cc. of *standard E* HCl were required to decolourize the solution.

1 cc. of E HCl = .031 grm. Na_2O .

$$22.6 \times .031 = .7006.$$

$$\frac{.7006}{1.25} = .5605 \text{ grm. } \text{Na}_2\text{O} \text{ as NaHO in 1 grm. sample.}$$

$$.5605 \times 100 = 56.1 \text{ per cent. } \text{Na}_2\text{O} \text{ as NaHO.}$$

The total Na_2O was 59.52 per cent.

Then—

$$59.52 - 56.1 = 3.42 \text{ per cent. } \text{Na}_2\text{O} \text{ as carbonate.}$$

57 to 60 per cent. of total soda, and 2 to 3 per cent. soda as carbonate are frequent proportions in the commercial article.

Assay of Soda Ash (Na_2CO_3).

Weigh out 10 grms. of sample, dissolve in 300 cc. of distilled water, filter into a graduated 500 cc. stoppered flask, wash the residue with distilled water until the washings are no longer alkaline, and dilute to 500 cc. at 15°C . The solution is then thoroughly mixed. 100 cc. (= 2 grms. sample) are accurately measured out and poured into a 200 cc. beaker, and 37 cc. of *standard* E H_2SO_4 gradually added from a burette; the solution is now boiled for five minutes with cover on to eliminate CO_2 , allowed to cool, a few drops of litmus solution added, and the free acid titrated by a standard E solution of sodic hydrate until the last drop produces a blue coloration. Note the volume required.

Example.—10 grms. of a sample of soda ash were treated as above; on titrating the free acid with standard E NaHO 6.0 cc. were required.

Then—

$$37 - 6 = 31 \text{ cc. of standard E } \text{H}_2\text{SO}_4 \text{ neutralized.}$$

$$1 \text{ cc. of E } \text{H}_2\text{SO}_4 = .031 \text{ grm. } \text{Na}_2\text{O.}$$

$$31 \times .031 = 0.961 \text{ grm. } \text{Na}_2\text{O} \text{ in 2 grms. sample.}$$

$$\frac{0.961 \times 100}{2} = 48.05 \text{ per cent. combined } \text{Na}_2\text{O.}$$

or,

$$\frac{31.41 \times 48.05}{31} = 48.7 \text{ English degrees.}$$

A great number of commercial soda ashes examined by the writer ranged from 46 per cent. Na_2O to 52 per cent. Na_2O .

The following is a *complete* analysis of Glasgow refined soda ash (*Brown*).

Sodium carbonates	80.92
„ hydrate	3.92
„ silicate	1.32
„ aluminate	1.01
„ sulphate	7.43
„ sulphite	1.11
„ thiosulphate	trace
„ sulphide	0.23
„ chloride	3.14
Insoluble matter	0.77
						<hr/> 99.85

PART VII.

GASWORKS PRODUCTS.

THIS section will include articles upon gas liquor, ammoniac sulphates, spent oxide, distillation of coal tar, and creosote for the pickling of timber.

Ammoniacal Liquor.

The ammoniacal liquor of the gas works, which derives its ammonia from the nitrogen present in the coal from which it is distilled, is a complicated solution containing a number of ammoniacal compounds, of which the following is a list :—

(a) VOLATILE AT ORDINARY TEMPERATURES.

Free ammonia.

Ammonium carbonates (mono, sesqui, and bi).

„ sulphide.
„ bisulphide.
„ acetate.
„ cyanide.

(b) FIXED AT ORDINARY TEMPERATURES.

Ammonium sulphate.

„ sulphite.
„ hyposulphite.
„ thiocarbonate.
„ chloride.
„ sulphocyanate.
„ ferrocyanide.

together with smaller portions of tarry matters, such as phenol, benzol, &c., pyridine, salts, &c.

Gas liquor is principally used for the production of sulphate of ammonia, much used as a manure, and its value for this purpose depends upon the proportion of total ammonia (free and combined) that it contains. Of the total ammonia from the liquor produced from English coals 15 to 20 per cent. is in the "fixed" state.

The following interesting table by Gerlach shows the amount of total ammonia contained in the ammoniacal liquors from various German and English coals:—

Coal Distilled.	grms. NH_3 per 100 cc.	degs. Baumé.	Gas Works.
<i>German Coals.</i>			
Zwickau coal and Bohemian lignite .	0.908	1.66	Leipzig
Zwickau and Burgk coal	0.944	1.5	Dresden
Burgk coal	1.862	2.8	Freiberg
Ruhr coal	1.812	2.1	Bonn
"	1.292	1.75	Eupen
"	1.826	2.25	Cologne
"	1.278	1.5	Dortmund
Saar coal	1.574	—	Augsberg
"	1.523	2.5	Treves
"	1.870	3.2	Zurich
Silesian coal	2.339	3.0	Berlin
" "	1.290	—	Pest
" " and Bohemian lignite .	1.030	2.0	Prague
<i>English Coals.</i>			
Cannel coal : boghead	2.881	3.75	Hamburg
English coal	3.514	4.0	Stettin
" "	2.659	3.0	St. Petersburg
" "	2.244	3.3	Trieste
" "	2.142	3.5	Stralsund
Leverson Wallsend	2.366	4.0	Altona
English coal	2.407	4.0	Copenhagen
New Pelton : Ravensworth . .	1.785	2.75	Riga
Old Pelton Main	1.717	3.0	Königsberg
English coal	2.966	3.0	Stettin
Old Pelton Main : Leverson Wallsend	1.345	3.0	Dantzic

The Valuation of Ammoniacal Liquor.—We shall here only make a determination of the free and combined ammonia with

the amount of lime required and its cost for expelling the combined ammonia. A method much used for valuing liquors is to obtain the specific gravity of them, and by referring to a table the percentage of ammonia is obtained; but owing to the difference in density of the various ammonium salts present, this method might be considered practically worthless. The following table by Siedel shows the differences in the percentage of ammonia in different liquors of the same specific gravity at 15° C. :—

Degrees, Baumé .	2	2.5	3	3.5	4	4.5	5	6
Specific gravity . .	1.0138	1.0163	1.0208	1.0249	1.0280	1.0316	1.0352	1.0426
Per cent. NH ₃	1.16 1.42 1.50 1.77	1.30 1.43 1.63 1.77 1.98 2.18 2.65	1.63 1.76 1.90 2.10 2.38 2.45	1.87 2.00 2.24 2.40 2.72	2.55 2.72 2.90 3.40	2.79 2.85 3.06 3.40 3.53	3.67	3.74

The following is a good method for the determination of the amount of the free and combined ammonia present in gas liquors. The specific gravity is first taken at 15° by hydrometer.

Determination of "Free" Ammonia.—Accurately measure out 20 cc. of the sample, pour into the flask A, Fig. 30, holding about 300 cc., dilute with about 100 cc. of distilled water, fix to condensing arrangement as shown in the sketch, and keep the tap B closed. E is a tube filled with glass wool, and serves to trap any traces of ammonia that might otherwise escape. When all is ready fixed for distillation, 30 cc. of standard E H₂SO₄ are run into D through the tube E, followed by a few drops of litmus solution. The glass wool will thus be

coloured red. If any of the ammonia escapes (which rarely happens) during the distillation, the stratum of glass wool would be completely coloured blue. Apply a gentle heat to A until it gently boils, and keep at this temperature until about 60 cc. have collected in D. The tap B is now opened and all acid washed out of E into D. It now becomes necessary to ascertain how much of the acid has been neutralized by the "free" ammonia distilled off; for this purpose detach D, gently boil

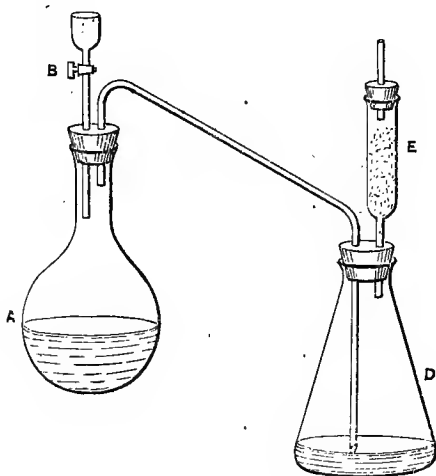


FIG. 30.

to eliminate any CO_2 , fill a burette with *standard* E sodic hydrate, and run this into it gradually until the last drop added turns the solution from red to blue. For every cc. of E H_2SO_4 neutralized by the sample there is .017 grm. of NH_3 in solution.

Combined Ammonia.—The flask D is now swilled out and again attached to E and A, and 20 cc. of standard E H_2SO_4 coloured with litmus are poured through E into D; 40 cc. of E NaHO (recently boiled to eliminate any NH_3 present) are

now run into the residual solution in A through B, and the distillation proceeded with as before, the solution being reduced nearly to dryness. The free acid in D is titrated with standard E NaHO as before. The number of cc.'s of acid neutralized $\times .017 =$ grms. of combined ammonia in 20 cc. of sample.

Example.—A sample of ammoniacal liquor had a specific gravity of 1.026 at 60° F.

Free Ammonia.—20 cc. of sample were taken, diluted, and distilled into 30 cc. of standard E H_2SO_4 . On titration with E NaHO 11.6 cc. were required to produce blue tint.

$$30 - 11.6 = 18.4 \text{ cc. E } H_2SO_4 \text{ neutralized.}$$

$$1 \text{ cc. of E } H_2SO_4 = .017 \text{ grm. } NH_3.$$

$$18.4 \times .017 = .3128 \text{ grm. } NH_3 \text{ in 20 cc.}$$

$$20 \text{ cc. of liquor} = 20 \times 1.026 \text{ (sp. gr.)} = 20.52 \text{ grms.}$$

$$20.52 : 100 :: .3128 : x = 1.5244 \text{ per cent. "free" ammonia.}$$

$$1 \text{ gallon of liquor weighs } 10 \times 1.026 = 10.26 \text{ lbs.}$$

$$103 : 10.26 :: 1.5244 : x = 0.1564 \text{ lbs. free } NH_3 \text{ per gallon.}$$

Combined Ammonia.—The residual solution from the estimation of the "free" ammonia was distilled with sodic hydrate into 20 cc. of standard E H_2SO_4 , and on titrating the excess of acid back with E NaHO, 11.7 cc. were required to produce blue tint.

$$20 - 11.7 = 8.3 \text{ cc. E } H_2SO_4 \text{ used.}$$

$$8.3 \times .017 = .1411 \text{ grm. combined } NH_3 \text{ in 20 cc. (= 20.52 grms.) of liquor.}$$

$$20.52 : 100 :: .1411 : x = 0.6876 \text{ per cent. combined ammonia.}$$

TOTAL AMMONIA.

$$1.5244 + .6876 = 2.212 \text{ per cent. total ammonia.}$$

$$100 : 10.26 :: 2.212 : x = .22695 \text{ lbs. of total } NH_3 \text{ in a gallon of liquor.}$$

Now every 34 parts of ammonia (NH_3) form 132 parts of ammoniac sulphate $(NH_4)_2SO_4$ then

$$34 : .22695 :: 132 : x = 0.8811 \text{ lb. of } Am_2SO_4$$

could be produced from one gallon of liquor ; and $\frac{2240}{.8811} = 2542$

gallons of liquor required, to produce one ton of pure sulphate of ammonia.

Quantity and price of lime required to expel the combined ammonia from 1,000 gallons of liquor. Say 90 per cent. lime is 22s. per ton.

Now every 34 parts of combined ammonia require 56 parts of pure lime (CaO) for its expulsion.

1,000 gallons of the liquor weigh 10,260 lbs., and the number of lbs. of combined ammonia in 1000 gallons is—

$$100 : 10,260 :: .6876 : x = 70.55 \text{ lbs.}$$

$$\therefore \text{ lbs. of pure lime required} = \frac{70.55 \times 56}{34} = 116.2 \text{ lbs.}$$

$$\begin{aligned} \text{,, } 90\% \text{ ,, } &= \frac{100 \times 116.2}{90} = 129 \text{ lbs.} = 1s. 4d. \\ &\text{per 1,000 gallons.} \end{aligned}$$

Assay of Sulphate of Ammonia (Am_2SO_4).

The value of commercial sulphate of ammonia in this country, is deduced from the percentage of ammonia present, and in Germany from the percentage of nitrogen. The pure salt contains 21.21 per cent. of nitrogen, equivalent to 25.75 per cent. of ammonia (NH_3). The specification for sulphate in this country enacts that it should contain 23 per cent. of combined ammonia; but the *commercial* article often contains between 24 and 25 per cent. The analysis of sulphate of ammonia should include the determination of water, combined ammonia, free sulphuric acid, and any sulphocyanates.

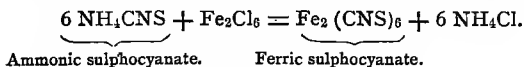
Estimation of Water.—10 grms. of the sample are weighed into a tared beaker and dried in the water oven until no further loss occurs; the loss is taken as water.

Estimation of Free Sulphuric Acid.—Weigh out 5 grms. of the sample into a 150 cc. beaker, dissolve in 100 cc. of distilled water, boil for a few minutes to expel any CO_2 , cool, add a few drops of litmus solution, and run in standard E sodic hydrate from a burette until the last drop added turns

the solution from red to blue. Note volume required. Each cc. of E $\text{NaHO} = \cdot 049$ grm. of free sulphuric acid present.

Estimation of Combined Ammonia.—The method adopted for the estimation of the combined ammonia in gas liquor is suitable for its determination in sulphates. 10 grms. of the averaged sample are weighed into a beaker, dissolved in water, diluted to 250 cc. at 15°C. , and thoroughly mixed. 50 cc. of this solution ($= 2$ grms. sample) are now poured into the flask A, Fig. 27, and 35 cc. of standard E H_2SO coloured with litmus, added through E into the flask D, and attached ready for distillation. 5 grms. of sodic hydrate dissolved in 50 cc. of water and boiled to expel any NH_3 present, are now poured into A through the tap B, which is afterwards closed, and the distillation proceeded with, which should take about two hours, the solution being prevented from getting too low in A by frequent additions of water through B. After it is assured that all the ammonia has been expelled, the tube E is washed into D, and the excess of acid therein titrated with standard E NaHO ; each cc. of acid neutralized $= \cdot 017$ grm. of ammonia.

Estimation of Sulphocyanates.—Owing to defective manufacture, small quantities of sulphocyanic acid often find their way into the finished product, which is supposed to have a very detrimental influence on plant life, and thus materially diminishes its value as a manure. A rough method much used for testing for sulphocyanates, is to place a small heap of 5 grms. of the sample on a sheet of white paper, and drop on it a solution of ferric chloride until the whole becomes saturated with it, when, if any sulphocyanates be present, a more or less red coloration will be produced, owing to the formation of ferric sulphocyanate. The following reaction takes place:—



If a similar heap of pure sulphate of ammonia, free from sulphocyanate, be placed by the side of the sample, and saturated with ferric chloride solution, a solution of potassic

sulphocyanide of known strength can be gradually run on to it, and stirred with a glass rod until it is seen that the tints are near alike; a rough idea is thus formed of the amount present.

The proximate estimation of sulphocyanate in commercial sulphate of ammonia is attended with difficulties, owing to the small quantity usually present, and to the influence of other complicated substances on its estimation by the usual processes. With care, the following colorimetric process based on the above reaction, will give fair results :—

Weigh out 4 grms. of the sample, dissolve in a little water, dilute to exactly 14 cc., filter off 7 cc. (= 2 grms. sample), into a carbon tube (Fig. 5), add 0.5 cc. of 5 E H_2SO_4 , and 1 cc. of 5 E Fe_2Cl_6 , and thoroughly mix. It now becomes necessary to obtain a standard ferric sulphocyanate solution. Owing to the peculiar tint of a solution of the commercial sulphate, neither an aqueous nor a pure ammoniac sulphate solution of the same strength will answer for comparison; the best method the writer knows of, is to make a *standard* of some of the sample, in which the sulphocyanates have been previously eliminated by evaporating to complete dryness with sulphuric acid. Weigh out 4 grms. of the sample into a small porcelain dish, mix with 1 cc. of 5 E H_2SO_4 , and dry in a water oven for a couple of hours. By this means the sulphocyanates will be practically all gone. Dissolve in water, dilute to 10 cc., filter off 5 cc. into a carbon tube, add 1 cc. of 5 E Fe_2Cl_6 , and dilute to within 2 cc. of the volume occupied by the sample; now run in 0.2 cc. at a time, from a 2 cc. pipette, graduated in 0.05 cc.'s, a solution of $\frac{\text{E}}{50}$ potassic sulphocyanate, until the tints are nearly alike, finally adjusting the volumes with water.

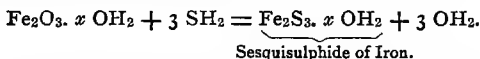
$$1 \text{ cc. of } \frac{\text{E}}{50} \text{ KCNS} = .00116 \text{ gm. CNS.}$$

The following is an analysis, by the above processes, of a bad sample of sulphate :—

Water	3.50 per cent.
Combined ammonia	23.45 „
Free sulphuric acid	trace
Sulphocyanate (CNS)09 „
Corresponding to AmCNS12 „

Spent Oxide.

The important part played by hydrated peroxide of iron in the absorption of sulphuretted hydrogen and other detrimental constituents of crude coal gas, renders the residual spent oxide a valuable product. With sulphuretted hydrogen the following reaction occurs :—



On exposing the sesquisulphide of iron thus produced to the action of the air, peroxide of iron is again produced, while free sulphur is formed thus :—



and the mixture would be capable of absorbing a further quantity of SH_2 ; the exposure could be repeated several times, until the oxide is completely choked with sulphur.

When the spent oxide has completed its duty, it is left exposed to the atmosphere, with frequent stirring up, until completely revived; it is then assayed for sulphur and sold, the price varying according to the amount of sulphur present.

Estimation of Sulphur.—The recognised method for the determination of sulphur in spent oxide is very simple. All that has to be done is to dissolve the sulphur out of a known weight of the sample by means of pure redistilled disulphide of carbon, which is afterwards distilled in a weighed flask, and the residual sulphur weighed. The following is the manner in which the sulphur is determined: .5 grms. of the averaged sample are weighed into the tube A (Fig. 31), into which a

plug of glass wool is fitted in the constriction. It is fitted into the weighed flask B contained in a sand bath, and connected to the bottle C, containing pure disulphide of carbon, by the tube T; and to the condenser D, by means of the tube M. When all is fixed, CS_2 is made to percolate through the sample by blowing into C, through Q. The sulphur is thus dissolved out into B; when B is nearly full the Bunsen, F, is lighted, and the disulphide distilled off through D into the flask E, which is

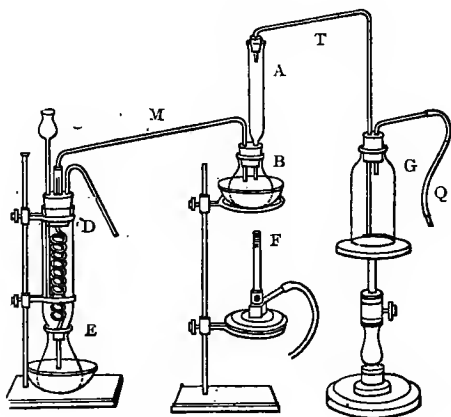


FIG. 31.

afterwards poured back into C, and the percolation continued several times, until it is assured that the whole of the sulphur is dissolved out. The CS_2 having been finally distilled, the residual sulphur is dried in the water oven and weighed; its weight $\times 20 =$ percentage of sulphur.

Synopsis of the Distillation of Coal Tar.—The following tabular statement shows the manner in which the various products from coal tar are obtained :—

Dehydration, *a.* by standing
b. during the heating up
Distillation. } **Ammoniacal
Liquor**

I. *Fraction up to 170° C.* { Ammoniacal liquor
 rectified yield :— { First runnings

- (1) Product up to 110° C., chemically washed
 Distilled by steam, yields, *a*
b. Weaker benzol goes to 1, 2 } **90 per cent. Benzol**
 (2) Product up to 140° treated like (1), yields *a* }
b. } **50 per cent. Benzol**
c. Intermediate fraction is redistilled
d.
 (3) Product up to 170° treated like (1) and (2) } **Solvent Naphtha**
 yields *a* }
b. } **Burning Naphtha**
c. Residue, goes to II.

II. *Fraction from 170° to 230°. Middle oil—*
 washed with caustic soda yields :—

- (1) Oil, distilled in the light oil still, yields—
a. Distillate up to 170° goes to (1), (3)
b. „ „ 230°, yields **Napthalene**
c. Residue goes to III.
 (2) Alkaline liquor decomposed by CO₂ yields :—
a. Aqueous solution of sodium carbonate,
 causticised by lime and used over again
b. Crude carbolic acid is purified and
 yields, *a* **Carbolic Acid**
β. Waste oils, go back to II.

III. *Fraction from 230° to 270° = Heavy oil*
 (collected till solid matters begin to crystal-
 lize).

Can be treated for naphthalene, usually
 only employed as } **Creosote Oil**
 or else separated into *a* }
b **Lubricating Oil**

IV. *Fraction, anthracene oil*

Filtered or cold-pressed yields :—

- (1) Oils, are redistilled and yield
a. Solid distillate, treated along with IV. (2)
b. Liquid distillate, goes to III. *b.*, or is re-
 distilled.
c. Residue (pitch, coke, &c.)

- (2) Residue is hot pressed and yields
a. Oils, treated like IV. (1)
b. Crude anthracene, washed with naphtha,
 &c., yields, *a* **Anthracene**
β. Solution is distilled, and yields,
aa. Naphtha used over again for washing
bb. Phenanthrene, &c., is burnt to . **Lampblack**

- V. Pitch. Employed for patent fuel or varnishes,
 &c. **Pitch**
 or else distilled, yielding :—
 (1) Crude anthracene, treated like IV. (2)
 (2) Lubricating oil, goes to III. *a*, resp. III. *b*
 (3) Residue **Coke**

The following table shows the yields, in percentage, of the various products from different tars (Calvert).

	Light oil.	Neutral heavy oil.	Phenol.	Paraffin.	Naphthalene.	Pitch.
Boghead . .	12	30	3	41	—	14
Cannel coal .	9	40	14	—	15	22
Newcastle coal	2	12	5	—	58	23
Staffordshire coal	5	35	9	—	22	29

Creosote as used for the "Pickling" of Timber.

The following is an extract from an interesting report which was made by Dr. Tidy to the Directors of the Gaslight and Coke Company, in August, 1883,* upon this subject :—

"The advantages to be derived from the creosoting process are of a threefold nature :

"1st. *A physical action.*—A very great increased solidity is effected by choking up the pores, thus agglutinating the whole mass of the wood into a more or less solid block. Apart from its rendering the wood more solid, this physical action is important in preventing the subsequent absorption of moisture.

* *Vide* "Lunge's Coal Tar and Ammonia."

“2nd. *A physiological action.*—The smell of creosote imparted to the wood prevents germinal life, well known to be destructive to timber, from being developed within it. Seeing that the preservation of timber has been effected by such materials as chloride of zinc, sulphate of copper, &c., with greater or less success, and that the action of these bodies must be mainly—although I admit not entirely—dependent on their toxic properties, this physiological action is one of importance. It must be remembered, however, that creosote has the advantage of a well-marked smell, which odour most of the lower animals dislike. In this respect it is superior to the other bodies I have named.

“Further, it is worth pointing out that all the constituents of the coal tar, and not the tar acids only, have a more or less well-marked tarry odour.

“3rd. *A chemical action.*—Respecting the chemical action I would draw attention to the fact that tar acids are not only antiseptic, but that they possess the power of coagulating albumen. It is to this latter action that I shall have to refer later on in this report, as playing an important part, in my opinion, in the preservation of timber.

“Now the following questions arise:—(1st.) Upon what constituents of the creosote does its value specially depend, and what are the relative values of its different constituents? (2nd.) If there be constituents in the creosote, which of themselves possess no special value, do they in any respect lessen the activity of the valuable constituents?

“The importance of considering the precise value of the several constituents of creosote arises as follows:—

“Speaking generally, creosote may be divided into two classes, London and Country creosotes. By London creosote we mean the creosote derived from the tars of London gasworks, the east coast generally, and from the gasworks of towns such as Southampton, Brighton, &c., where the coal employed is Newcastle coal. So far as I am able to learn, the larger proportion of the creosote produced in England is of this character. The two creosotes being very different in

their composition, it becomes important to consider them separately.

"*The London Creosote* has a somewhat high specific gravity, and contains a comparatively large percentage of naphthalene, and a small percentage (*i.e.*, less than 10 per cent.) of tar acids. Further, it contains a considerable quantity of the heavier portions of the oil, that is, of those portions not volatile at a temperature below 316° C.

"*The Country Creosote*, on the other hand, has a less specific gravity, and is considerably more fluid than London creosote. It contains considerably less naphthalene than the London creosote, a larger total percentage of tar acids, and a smaller percentage of the heavier portions of the oil present.

"The real question I have had in view in this inquiry being Country creosotes *versus* London creosotes, it became necessary to inquire into the relative values of the heavier portions of the oil, of the naphthalene, and of the tar acids in creosoting.

"The tar acids, in the first instance, effect the coagulation of the albumen of the wood sap. This coagulated albumen mixes with the naphthalene of the creosote, which, so soon as the temperature becomes sufficiently reduced, is redeposited, and forms, along with the heavier portions of the oil, a solid magma within the pores and fibres of the wood. That this formation of solid magma actually occurs, I have convinced myself by numerous microscopic examinations of creosoted timbers.

"The success of the process, therefore, being presumably assisted by the coagulation of the albumen, the question arises, *What quantity of tar acids is necessary to effect this object?*

"There is little doubt in my mind that 2 or 3 per cent. would amply suffice to effect this coagulation of the sap albumen.

"We are now led to consider if any value and, if any, what value is to be ascribed to the tar acids beyond that needed to effect the coagulation of the albumen.

"I am far from prepared to say they are otherwise entirely valueless. Still it is a remarkable fact, which I have over and

over again verified, that in the timbers that have been creosoted for a considerable time (say a year), very small quantities indeed (if any) of the free tar acids are to be found.

"I have upon this point instituted a series of examinations of sleepers, obtained from independent sources, and of ages varying from one to twenty years; and it is a fact worth noting that, within a very short time after a sleeper has been in use, the tar acids appear to be entirely dissipated.

"Seeing however that the life of a sleeper is by no means so limited, the facts I have mentioned suffice to show that the action of the tar acids *per se* cannot have any great or permanently preservative influence in creosoting.

"I admit it was natural to suppose that bodies commonly regarded as powerfully antiseptic should have been the active agents in the process. Further, I must admit that it was with such view I commenced this inquiry. My recent investigations, however, have clearly shown that the value of the tar acids in the creosoting process has been greatly over-estimated.

"I am convinced that, so long as the quantity of carbolic acid present in the creosote is sufficient to coagulate the albumen of the wood sap, it is also sufficient for practical purposes.

"I have now to consider the value of naphthalene.

"I am disposed to think that this body is of infinitely greater value than at first sight appears. Admitting that as an antiseptic it is inferior to the tar acids, nevertheless, so far as preservative action alone is concerned, it must not be supposed to be inoperative. Its special value, however, consists in helping to render the wood solid.

"But it may be said, granting this to be the case, naphthalene is so volatile, that the heat of the sun, especially the intense heat of an Indian climate, would soon drive the whole of it off. It is true that on exposing a block of creosoted timber in an oven to a temperature of 54.5° C., and this may be taken to be an extreme tropical heat, the door of the oven after a short time shows conclusively that some of the naphthalene in the sleeper has undergone volatilization by the heat applied.

"I would, however, direct attention to the following experiment:—

"I exposed a large block of creosoted timber (accurately weighed) to a temperature of 65.5° C. On weighing this at the end of twenty-four hours, I found it to have lost 1,200 grains. On exposing the same block to the same temperature for another twenty-four hours, it lost 135 grains; whilst on continuing the experiment for a third twenty-four hours, it lost only 15 grains. After this the loss was practically nil.

"I now planed off about a quarter of an inch of the block I had already heated. This done, I again exposed it to a heat of 54.5° C. for twenty-four hours, during which time it lost 1,150 grains. The loss on the second day was less than 100 grains, whilst on the succeeding days the loss was practically nil.

"The surface of the wood was again planed off, and similar experiments repeated a third time, with almost identical results.

"From numerous microscopical examinations of the timber, and from the experiments I have described, I consider that I am justified in drawing the following conclusions *re* naphthalene:—

"1st. That supposing, for the sake of argument, naphthalene possesses no great antiseptic power, nevertheless it acts beneficially by clogging up the pores of the wood, forming a more or less solid magma with the coagulated albumen. In this way it assists the physical part of the creosoting process, upon which the preservation of timber materially depends.

"2nd. That although a certain quantity of naphthalene would undoubtedly be volatilized by a tropical heat, nevertheless the loss would be practically limited to the *surface* of the timber, and would be complete a day or two after the exposure, the naphthalene in the deeper parts of the wood remaining fixed by incorporation with the albumen coagulated by the action of the tar acids.

"3rd. That inasmuch as the naphthalene cannot injure the action of the tar acids, or other constituents of the creosote, and is itself a positive benefit to the process, there is not

only no object in requiring that the oil used for creosoting should be free from naphthalene, but that it would be unadvisable to demand such freedom.

"There are many other facts that in my judgment corroborate the views I have expressed. Thus I am given to understand that, during the twelve years after the process of creosoting was first introduced into India, the whole of the sleepers were prepared with heavy London creosote (that is, a creosote heavily charged with naphthalene), with the occasional admixture of a small quantity of Country oil for the purpose of dilution.

"It is perfectly certain, further, that it was on account of the good results so obtained that creosoting became a process of acknowledged utility.

"So far as I can learn it was not until the Country oils became more extensively used that any complaints respecting the inefficiency of the process arose. From independent inquiries, I think there is the strongest possible reason to believe that the sleepers that proved unsatisfactory had been prepared with Country and not with London oil.

"Nothing has impressed me more strongly in the course of these inquiries than the value of the heavy oils present in the creosote, that is, of the oils that do not distil over under 316° C. Of a certain antiseptic power, and very difficult of volatilization, they are, I believe, bodies of great value in the oil employed in the creosoting process."

Dr. Tidy's Specification for Creosote.

Taking into consideration the arguments of the report just quoted, Dr. Tidy has recommended the following as a specification for creosote (amended in 1885):—

"(1) That the creosote shall be completely liquid at a temperature of 38° C., no deposit afterwards taking place until the oil registers a temperature of 35° C.

"(2) That the creosote shall contain at least 25 per cent. of constituents that do not distil over at a temperature of 316° C.

"(3) That tested by the process hereafter to be described, the creosote shall yield a total of 8 per cent. of tar acids.

"(4) That it shall contain no admixture of bone oil, shale oil or of any substance not obtained from the distillation of coal tar, and that the first 25 per cent. of the distillate shall have a specific gravity greater than that of water.

Process to be adopted for determining the coal tar acids,

"(1) 100 cc. of the well mixed creosote are to be distilled at a temperature of 316° C. until no further distillate comes over. The distillate so obtained is to be mixed and well shaken in a stoppered flask with 30 cc. of a solution of caustic soda, sp. gr. 1.2 (5 E). The mixture is then to be heated. This done, the stopper is to be replaced in the flask, and the hot mixture again shaken vigorously for at least a minute.

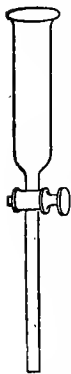


FIG. 32.

"The contents of the flask are now to be poured into a separating funnel (Fig. 32), and the soda solution drawn off. The creosote is to be heated a second and a third time in a similar manner with the caustic soda solution, except that only 20 cc. of the soda solution shall be used for the second and third extraction, instead of 30 cc. as in the first extraction.

"(2) The three soda solutions are now to be mixed together. *When cold*, any particles of creosote are to be got rid of by means of a separating funnel. This done the solution to be thoroughly boiled, in order to expel the last traces of creosote present in the solution. The mixture is then allowed to be cooled. When cold 9 E H_2SO_4 is to be added (about 35 cc. will be required) until the solution becomes slightly acid to litmus. The whole is then to be poured into a separating funnel, and allowed to stand until perfectly cold and the tar acids well separated.

"(3) The tar acids are now to be dissolved in 20 cc. of the caustic soda solution, sp. gr. 1.2 (5 E), and 10 cc. of water. The mixture is then to be boiled and filtered through a funnel

fitted with a plug of asbestos. The asbestos is to be washed with not more than 5 cc. of boiling water. The solution is to be allowed to cool *perfectly* in a 100 cc. measure. It is then to be rendered slightly acid, with 12 E H_2SO_4 (10 cc. will probably be found sufficient for this purpose). The whole is again allowed to stand for *two hours* until *perfectly cold*, when the percentage of the tar acids is to be read off.

Process to be adopted in estimating quantity of distillate.

"The operation is to be conducted in a retort (fitted with a thermometer), and heated with the naked flame of a Bunsen burner. The heat applied is to be gradually raised to 316°C. , and continued at that temperature until no further matters distil over. The distillation of the 100 cc. should be completed within half an hour."

Sir F. Abel's Specification for Creosote.

The following is a specification for creosote which was drawn up by Sir Frederick Abel in January, 1884:—

"The creosoting liquor is to be of the description known as heavy oil of tar, obtained by the distillation of coal tar, and consisting of that portion of the distillate which comes over between the temperature of 176°C. and 405°C.

"The liquor must be free from admixture with any oil or other substance not obtainable from such distillate. It shall contain not less than 20 nor more than 30 per cent. of constituents that do not distil over at a temperature approaching 316°C.

"It must yield not less than 9 per cent. by volume of tar acids.

"The creosoting liquor must become completely fluid when raised to a temperature of 38°C. , and exhibit no signs of any deposit on cooling down to a temperature of $32\frac{1}{2}^\circ \text{C.}$

"The specific gravity of the liquor must not be less than 1.035, and not more than 1.065, at a temperature of 32°C. , as compared with water at $15\frac{1}{2}^\circ \text{C.}$ "

Creosote Oils.—The following table shows the character of various creosote oils* :—

	Specific Gravity at 32° C.	Per cent. of Distillate below 315° C.	Per cent. of Tar Acids from Distillate.
<i>A.</i> Heavy London oils : highest	1·075	79	8·0
Ditto lowest	1·048	60	3·0
Average of 20 samples . .	1·0588	71·5	5·6
<i>B.</i> Partial runnings from London oils : highest . .	—	91	10·2
Ditto lowest . .	—	78	8·2
Average of 20 samples . .	—	82·8	91·5
<i>C.</i> English Country oils : highest	1·056	90	24·0
Ditto lowest	1·024	72	13·5
Average of 18 samples .	1·0335	81·8	18·6

Carbolineum.—This is a substance becoming much used as a timber preservative. It is, however, mostly used superficially for painting telegraph poles, wood pavements, fences, &c., and is said to effectually prevent water from getting into the wood. It has a remarkably high boiling point, and is prepared from the anthracene oil, squeezed out of rough anthracene, which is mixed with sulphate of copper or chloride of zinc, and certain proportions of boiled linseed oil or resin. Owing to the absence of phenol, it may not be considered so effectual for the pickling of sleepers in the ordinary way as creosote would be.

* Allen's "Comcl. Org. Anal.," V. ii.

PART VIII.

DISINFECTANTS.

THE assay and valuation of the disinfectants treated of here, will include those that are, commercially speaking, the most in use, viz., carbolic acid, carbolic acid powders, chloride of lime (bleaching powder), and permanganate of soda or potash (Condy's Fluid).

carbolic Acid (Phenol, C_6H_5HO).

Carbolic acid, which is a powerful antiseptic and disinfectant, is principally obtained from that fraction obtained on the distillation of coal tar which distils between 150° C. and 200° C. In the pure state it is a colourless crystallized solid, melting at 42.2° C., and boiling at 182° C. A commercial carbolic acid much used as a disinfectant is a dark-coloured liquid with a characteristic tarry smell, ranging in specific gravity from 1.04 to 1.06, and sometimes containing a considerable proportion of neutral tar oils of little use as a disinfectant.

Absolute crystallized phenol is miscible with water at the ordinary temperature, to the extent that the most concentrated solution contains about 73 per cent. of the pure acid, while the liquid thus produced is soluble in about 11.2 times its measure of water at 15° C. The solubility, however, is greatly increased at

higher temperatures, being miscible in all proportions with water at about 73° C. The pure acid is miscible in all proportions in benzol, ether, carbon disulphide, acetic acid (17 E), alcohol, and glycerine.

Carbolic acid acts as a powerful disinfectant and antiseptic owing to its property of coagulating albuminous substances, and preventing germinal life from propagating. A dilute solution of phenol is the most satisfactory agent we know of for arresting and preventing mortification.

By determining the neutral tar oils in a known volume of a sample of commercial carbolic acid, and subtracting the amount thus found from the volume of the original sample taken, an *approximate* idea of the value of a sample could thus be obtained.

Estimation of Neutral Tar Oils.—Measure out 5 cc. of the crude acid into a carbon tube, Fig. 5, add to this 10 cc. of a 2 E solution of sodic hydrate, and thoroughly shake for some time; the soda thus combines with the tar acids, forming phenate and cresylate of soda, &c., which is soluble in the aqueous solution. On allowing the mixture to stand some time, the neutral tar oils will settle out, sinking to the bottom or floating on the top, according to whether they are heavier or lighter than the aqueous liquid; 5 cc. of petroleum ether are now added, the mixture thoroughly shaken, the volume of the ethereal solution of the neutral oils is read off, and on subtracting the 5 cc. of added ether, the remainder is equal to the volume of neutral oils present in the 5 cc. of sample, which multiplied by 20 gives the percentage.

Estimation of Phenol and Cresol.—These are the principal acting disinfectants in commercial carbolic acid, and Dr. Tidy asserts that cresol (C_7H_7OH) is as equally efficacious as pure phenol (C_6H_5HO). If the amounts of these be required collectively, the volumetric process devised by Koppeschaar, depending upon the formation of tri-bromo-phenol ($C_6H_2Br_3OH$), and tri-bromo-cresol ($C_7H_4Br_3OH$) gives very fair results. The following are details of the process:—

Solutions required.—A solution of thiosulphate of soda

($\text{Na}_2\text{S}_2\text{O}_3$) of such strength that 1 cc. of it would be theoretically equivalent to .005 gm. of iodine, is prepared by dissolving 9.764 grms. of crystallized thiosulphate of soda ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$) (previously dried between blotting paper) in distilled water, and diluting to 1,000 cc. at 15°C . It should be standardized by the method herein described with a solution of iodine containing 5 grms. per litre.

Starch Solution.—Weigh out 1 gm. of starch, make into a thin paste with cold water, pour on 100 cc. of boiling water, stir, allow sediment to subside, and bottle the clear supernatant liquid.

Bromine Water.—Dissolve about $1\frac{1}{2}$ grms. of liquid bromine in 1 litre of distilled water.

Potassium Iodide Solution.—Dissolve 125 grms. of pure KI in water, and dilute to 1,000 cc.

When all the above solutions have been made, dissolve 4 grms. of the sample in water, dilute to 1,000 cc. at 60°C . in a graduated stoppered flask, thoroughly mix, and put on one side while the bromine solution is valued as follows:—Fill a Mohr's burette with the standard thiosulphate solution, then measure off accurately 50 cc. of the bromine solution into a 100 cc. beaker and add 5 cc. of the potassic iodide. Iodine is thus liberated; now run in gradually the thiosulphate solution, stirring till the violet colour of the iodine has nearly all disappeared (the reaction here is:— $2 \text{Na}_2\text{S}_2\text{O}_3 + 2 \text{I} = 2 \text{NaI} + \text{Na}_2\text{S}_4\text{O}_6$), then add 2 cc. of starch solution, when the blue colour of iodide of starch will be produced with the small quantity of free iodine still in solution; continue running in the thiosulphate solution until the last drop added completely decolourizes the solution. Note the volume required. 25 cc. of the solution of the sample (= 0.1 gm. original sample) are now poured into a graduated stoppered 500 cc. flask, and diluted quickly up to the mark with the bromine solution (= 450 cc.). Fix stopper, thoroughly shake for about ten minutes, and allow to stand for about half an hour, after which pour out into a litre beaker, containing 5 cc. of the iodide solution. Stir and titrate with the standard thiosulphate solution in the same

manner as with the bromine solution, and note volume required.

From the following formula the percentage of carbolic and cresylic acids expressed as carbolic acid is obtained :—

Let X = thiosulphate required for 50 cc. bromine solution.

„ Y = „ „ for excess of bromine in the 25 cc. of the sample (= .1 grm.).

Then $(9.5x - y) 0.61753$ = percentage of carbolic acid.

Carbolic Acid Powders.

The best carbolic acid powders in the market are those which have for a base or absorbent, for the crude carbolic acid, substances which do not chemically combine with the carbolic acid, such as kaolin, calcium sulphate, kieselguhr, &c. Carbolic acid powders made with slaked lime appear to be of little use as disinfectants, since the carbolic acid thus combines with the lime, forming carbolate of lime. “Macdougall’s Disinfecting Powder” contains a crude sulphite of lime as a base. “Calvert’s Carbolic Acid Powder” is made by mixing the carbolic acid with the siliceous residuum left in the manufacture of alum from shale. There are five qualities made, containing 5, 10, 15, 20, and 50 per cent. of the crude acid.

Commercial carbolic acid powders may be considered of very fair quality if they contain from 15 to 20 per cent. of crude carbolic acid.

Method of Assay.—There are two methods in use for the assay of carbolic acid powders: (1) by distilling a known weight direct in a retort with the collection and examination of distillate, &c., a process applied to powders which contain the acid in a free state; and (2) liberation of combined acid with sulphuric acid and its subsequent extraction with benzol, &c.

(1) *Method for Powders containing Free Carbolic Acid.*—Weigh out 100 grms. of the averaged sample, transfer into a 150 cc. retort, attach a 100 cc. graduated tube as a condenser, apply a gentle heat at first to the retort with the naked

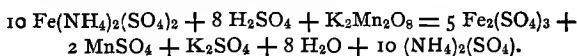
flame of a Bunsen burner, and gradually increase the heat until the contents of the retort are at a dull red heat, and no more liquid is being distilled. Note the volume distilled, and the amount of water separated out. It is important that the oily layer should be tested for neutral tar oils, which are apt to be used as a useless adulterant. Pour the liquid into a separator, separate the oily layer, and estimate the tar oils as described on page 266, and, if necessary, the carbolic acid by the Koppe-schaar process.

(2) *Method for Powders containing Combined Carbolic Acid.*—This process can be applied with equal advantage to powders containing free carbolic acid without the addition of sulphuric acid. Weigh out 50 grms. of the sample into a 200 cc. glass mortar; mix with the aid of a pestle with 5 cc. of water, and then run in gradually 18 E sulphuric acid, stirring until a decided but slight excess is added; 50 cc. of water are now added, and the whole poured off into a capacious separator; 60 cc. of 90 per cent. benzol are now added, and the mixture thoroughly agitated for some time, to ensure the complete extraction of the crude carbolic acid by the benzol. It is then allowed to separate, the benzol solution distilled, and the residual crude carbolic acid thus isolated examined for tar oils and carbolic acid by the methods before mentioned.

Assay of Permanganate of Potash (Condy's Fluid).—The value of this useful disinfectant depends upon the amount of available oxygen present. The molecule of pure permanganate of potash ($K_2Mn_2O_8$) contains five atoms of available oxygen, capable of destroying by oxidation organic matter, germs of disease, &c., and in order to determine the amount of available oxygen present we must find out how much of a given substance capable of being oxidized by it to a definite form is required for a known weight. Pure crystallized ammonio-ferrous sulphate [$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$] is a salt well adapted for this purpose, and an $\frac{E}{10}$ solution is of convenient strength to use, which is prepared by dissolving 39.2 grms. of the salt

(previously dried between blotting paper) in recently boiled distilled water, and diluting to 1,000 cc. at 15° C.; 1 cc. of this solution corresponds to '0008 grm. of available oxygen.

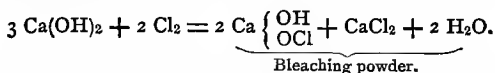
Five grms. of the sample are carefully weighed into a beaker, dissolved in recently boiled distilled water, diluted to 1,000 cc. at 15° C. in a graduated flask, thoroughly mixed, and any sediment present allowed to subside; 50 cc. of the clear solution (= '25 grm. sample) are measured into a 150 cc. beaker, and 20 cc. of 5 E H₂SO₄ added. A burette is now filled with the ammonio-ferrous sulphate solution, and this is gradually run into the solution of the sample, stirring until the last drop added gives a slight but permanent pink coloration to the liquid, which can be best observed by holding a piece of tissue paper behind the beaker. Note the volume required. The reaction that takes place is as follows:—



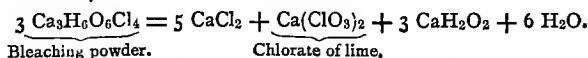
The number of cc.'s of $\frac{\text{E}}{10} \text{ K}_2\text{Mn}_2\text{O}_8$ required \times '0008 \times 4 \times 100 = percentage of available oxygen present.

Assay of Chloride of Lime (*Bleaching Powder*).—

This well known disinfectant is manufactured by passing chlorine gas over slaked lime *gradually*, so as not to raise the temperature of the substance, otherwise chlorate of lime will be produced, which is of no use as a disinfectant. The reaction which goes on when the temperature is kept low may be expressed as follows:—

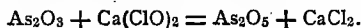


If the temperature be raised, it is decomposed with the formation of hydrate and chlorate of lime, and calcium chloride thus:—



The value of bleaching powder as a disinfectant, depends upon the liberation of hypochlorous acid, which is a very powerful oxidizing agent. The hypochlorous acid is brought about by the action of moist air containing carbonic acid, calcium carbonate being simultaneously produced. When used as a bleaching agent it is mixed with water; the article to be bleached is dipped into the mixture, and then into a dilute solution of sulphuric acid; chlorine is thus liberated in a nascent state in the fibre of the material, which combines with the hydrogen of water, thus liberating nascent oxygen, which in its turn destroys the colouring matter of the material. Good bleaching powder should contain about 35 per cent. of available chlorine, the maximum amount that can be present being about 39 per cent. But owing to careless manufacture, exposure to air, &c., the amount of available chlorine may be anything below this amount.

The best method for determining the amount of available chlorine in bleaching powder is that devised by Penot, which depends upon the oxidation of arsenious acid (As_2O_3) into arsenic acid (As_2O_5) by the sample, thus:—



Solutions Required.—*Deci-Normal Sodid Arsenite.*—Weigh out accurately 4.95 grms. of pure arsenious acid into a 300 cc. beaker, together with 25 grms. of pure crystallized carbonate of soda; pour on to the mixture 250 cc. of hot water, and heat and stir until all goes into solution; cool, pour into a graduated stoppered litre flask, dilute to 1,000 cc. at 15°C. , and thoroughly mix. 1 cc. of the $\frac{\text{E}}{10}$ solution = .00355 gm. chlorine.

Starch Test Papers.—Weigh out about 3 grms. of starch into a glass mortar, and make into a paste with about 50 cc. of water, with the aid of a pestle. Pour into a beaker and gradually add 200 cc. of boiling water, stirring, and then add 1 gm. of carbonate of soda and 1 gm. of potassic iodide dissolved in a little water, and dilute the whole to about 500 cc.

Strips of filter paper are moistened with this solution, and after carefully drying they are put into a stoppered bottle ready for use.

The Analytical Process.—10 grms. of the well mixed sample are weighed into a 150 cc. glass mortar, 60 cc. of water poured on it, the mixture thoroughly lixiviated with the aid of a pestle, and the sediment allowed to subside as far as possible; after which the supernatant and turbid solution is decanted off into a graduated stoppered litre flask; more water is added to the residue in the mortar and mixed as before, and the liquid poured off into the flask; the lixiviation with portions of water is repeated until the whole of the sample is thus got into the flask, and then finally diluted to 1,000 cc. at 15° C.

Accurately measure off 50 cc. of the solution (= .5 gm. original sample) into 200 cc. beaker, and gradually run in the standard $\frac{E}{10}$ sodic arsenite solution from a burette, stirring until a drop of the solution, taken out with the stirrer, no longer produces a blue stain of iodide of starch upon one of the prepared starch papers.

Example.—10 grms. of a sample of bleaching powder was treated as above, and 50 cc. (= .5 gm. sample) of the solution required 48.5 cc. of the arsenious solution to produce no further blue coloration of the starch paper.

$$1 \text{ cc. of } \frac{E}{10} \text{ arsenious solution} = .00355 \text{ gm. chlorine.}$$

$$48.5 \times .00355 = .1722 \text{ gm. chlorine in } 0.5 \text{ gm. of sample.}$$

$$.1722 \times 2 \times 100 = 34.44 \text{ per cent. available chlorine.}$$

PART IX.

EXPLOSIVES.

Nitro-Glycerine [$\text{C}_3\text{H}_5(\text{NO}_3)_3$].—Nitro-glycerine is prepared on a large scale by first mixing four parts of 36 E H_2SO_4 , and one part of 22 E HNO_3 (fuming) in a wooden vessel lined with lead, and allowing to cool, after which one part of glycerine is allowed to run into the mixture in a thin stream. On allowing to stand for some time, the nitro-glycerine separates as an oily liquid on the surface of the acid, which is separated, washed first with water, and finally with a dilute solution of sodic carbonate to eliminate the last traces of acids. It is important that all the free acid should be perfectly neutralized, since, if any is left in the finished product, it is liable to spontaneously decompose. The specific gravity of nitro-glycerine is 1.600 at 15°C ., and it solidifies at 8°C . When heated to about 257°C ., it decomposes with a sharp detonation; a similar result is obtained when a little of the substance is smartly struck or compressed. When ignited in air it burns with a greenish flame without explosion. According to Bloxam, when nitro-glycerine is dissolved in alcohol and warmed with ammonium sulphide it is decomposed, sulphur being separated, the reaction being:—



The value of nitro-glycerine can be determined from the amount of nitric oxide liberated when a weighed portion is treated with sulphuric acid over mercury in a nitrometer, as in the determination of nitrates in waters, page 156. This process was devised by Hempel and Lunge, who proceed as follows: 0.2 grm. is introduced into the cup of the nitrometer previously

filled with mercury, and 2 cc. of 36 E H_2SO_4 added, stirred with a glass rod, and the mixture made to run into the nitrometer. The cup of the nitrometer is rinsed out with successive small portions of 36 E H_2SO_4 , 3 cc. in all being used. The mixture is then thoroughly shaken up with the mercury until no further evolution of nitric oxide occurs. The number of cc's. of nitric oxide thus produced corrected to 0°C. , and 760 mm. mercury, multiplied by $\frac{3.39 \times 100}{200}$ gives the percentage of nitro-glycerine present.

Dynamite.—Ordinary dynamite contains 75 per cent. of nitro-glycerine and 25 per cent. of a porous siliceous substance, called kieselguhr. But there are numerous explosives of the dynamite class now in the market, containing a variety of substances, many of which are not classified among the explosives licensed in this country. For the composition of various kinds, see page 280.

Determination of Water.—Water cannot be determined by drying a weighed portion of the sample in a water oven, since nitro-glycerine is thereby volatilized. 2 grms. of the sample are reduced carefully, as finely as possible with the aid of a horn spatula, and dried in a vacuum over strong sulphuric acid until the moisture is eliminated.

Determination of Nitro-Glycerine.—When other substances soluble in methylated ether are absent, the nitro-glycerine can be determined by exhausting 2 grms. of the sample with methylated ether in a Soxlet's fat extraction tube (Fig. 33), and weighing the residuum, the loss being taken as nitro-glycerine. The nitro-glycerine cannot be accurately determined by the evaporation of the ethereal extract and weighing the residue, since there is a sensible loss of nitro-glycerine by this means.

The following is a tabulated scheme, arranged by Allen, for the analysis of nitro-glycerine compounds:—

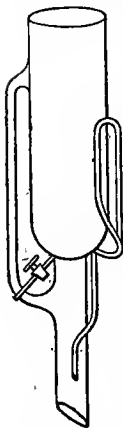


FIG. 33.

SCHEME FOR ANALYSING NITRO-GLYCERINE COMPOUNDS.

Exhaust the previously dried sample with anhydrous ether in a fat extraction apparatus.

Solution :—Divide into two equal parts.

Residue :—Dry, weigh, and exhaust with water in a Soxhlet tube.

A.—Allow the ether to evaporate at ordinary temperature, and dry the residue in vacuo over 36 E H₂SO₄ and weigh. Weight gives the sum of nitro-glycerine, resin, camphor (sulphur), and paraffin. Determine the nitro-glycerine by method given.

B.—Add a few drops of phenolphthalein and titrate with standard E alcoholic potash. 1 cc. of E KHO = 0.330 grm. resin. Add 10 cc. more E KHO and evaporate. Dissolve residue in water, shake with ether, and separate.

Solution :—Contains metallic nitrates, chlorates, soluble carbonates, &c., the sum of which (exclusive of ammonium carbonate) can be determined by evaporating the solution to dryness at 100° C., and weighing the residue. The nitrates can be here determined by the nitrometer.

Solution :—Evaporate and weigh residue, consisting of mono- and di-nitro-cellulose, collodion - cotton, which may be further examined in the nitrometer.

Residue :—Dry, weigh, and treat a weighed portion in nitrometer, calculated evolved gas to trinitro-cellulose, C₆H₇(NO₃)₃O₅. If present exhaust the remainder with acetic ether.

Ethereal solution evaporated leaves the paraffin.

Aqueous liquid :—Add bromine, acidify with 10 E HCl, separate any resin, and precipitate filtrate with E BaCl₂. BaSO₄ x .1373 = sulphur.

Solution contains trinitro-cellulose.

Residue :—Weigh, ignite, and re-weigh.

Loss equals cellulose, sawdust, charcoal, coal, &c.

Residue consists of kieselguhr, chalk, and other mineral substances.

Gunpowder.—Gunpowder is a mechanical mixture of nitre, charcoal and sulphur. Good charcoal obtained from dogwood, willow, or alder, is the best for the purpose; the sulphur preferred is that which has been distilled and ground to a fine meal; and nitre only of the first quality is employed.

The proportion of these ingredients varies in different countries, and also according to the purpose for which the gunpowder is proposed to be used.

The following table shows the percentage compositions of gunpowders of various countries :—

	Nitre.	Sulphur.	Charcoal.
English and Austrian (musket) .	75	10	15
Prussian (musket)	75	11·5	13·5
Swedish „	75	9	16
Chinese	75·7	9·9	14·4
French (musket)	75·0	12·5	12·5
„ (sporting)	76·9	9·6	13·5
„ (blasting)	62	20	18

The products of combustion of gunpowder are very complicated, the composition varying according to the manner in which the powder is fired. Karolyi, Noble and Abel succeeded in making an analysis of the products of combustion of gunpowder.

The following shows some of the results obtained by firing three kinds of powder under the conditions of artillery practice :—

(1) COMPOSITION OF THE POWDER USED.

	Ordnance powder.	Small arms powder.	Pebble powder (Noble and Abel).
Nitre	73·78	77·15	74·67
Sulphur	12·80	8·63	10·07
Charcoal. { Carbon	10·88	11·78	12·12
{ Hydrogen	0·38	0·42	0·42
{ Oxygen	1·82	1·79	1·45
{ Ash	0·31	0·28	0·23
Water	—	—	0·95
	99·97	100·05	99·91

(2) PRODUCTS OF COMBUSTION OF 100 PARTS BY WEIGHT.

	Ordnance powder.	Small arms powder.	Pebble powder (Noble and Abel),
Nitrogen . . .	9.77	10.06	11.39
Carbonic acid . . .	17.39	21.79	27.70
Carbonic oxide . . .	2.64	1.47	4.73
Hydrogen . . .	0.11	0.14	0.05
Sulph. hydrogen . . .	0.27	0.23	0.84
Marsh gas . . .	0.40	0.49	0.12
Total gaseous	30.58	34.18	44.83
Ammonic sesquicarbonate	2.68	2.66	0.04
Potassic sulphate . . .	36.95	36.17	6.58
„ carbonate . . .	19.40	20.78	30.98
„ thiosulphate . . .	2.85	1.77	3.38
„ sulphide . . .	0.11	0.00	10.55
Charcoal . . .	2.57	2.60	—
Sulphur . . .	4.69	1.16	3.40
Loss . . .	0.17	0.68	—
Potassic sulphocyanate . . .	—	—	0.13
„ nitrate . . .	—	—	0.11
Total solid	69.42	65.82	55.17

(3) PRODUCTS OF COMBUSTION BY VOLUME IN 100 OF GAS.

	Ordnance powder.	Small arms powder.	Pebble powder (Noble and Abel)
Nitrogen . . .	37.58	35.33	32.19
Carbonic acid . . .	42.74	48.90	49.82
Carbonic oxide . . .	10.19	5.18	13.36
Hydrogen . . .	5.93	6.90	2.08
Sulphuretted hydrogen . . .	0.86	0.67	1.96
Marsh gas . . .	2.70	3.02	0.58
	100.00	100.00	99.99

The Analysis of Gunpowder.—Estimation of Moisture.

—Weigh out 5 grms. of the powdered sample into a porcelain basin, and dry in *vacuo* over sulphuric acid until no further loss in weight occurs. The loss=moisture.

Estimation of Nitrogen—The residue from the estimation of

moisture is exhausted with water, and filtered through a tube containing a plug of glass wool, as in Fig. 1, into a weighed dish. When all soluble matter has been extracted, the solution is evaporated to dryness and the residue weighed. The residue should be again dissolved in water, and tested for sulphates and chlorides, the sulphates being precipitated with nitrate of barium, and the resulting BaSO_4 filtered off and estimated if necessary. The chlorides could be determined in the filtrate by precipitating with nitrate of silver, and filtering the resulting AgCl .

Estimation of Sulphur.—The residue left in the tube after the exhaustion with water, is dried in the tube by passing a current of air through, which has been previously dried by passing through sulphuric acid. The sulphur is then estimated in the manner described for the estimation of sulphur in spent oxide, page 253.

The Charcoal may be taken by difference. If it be necessary to determine the quality of the charcoal, this could be obtained by the method given on page 104 for the analysis of coal.

Subjoined are the compositions of a few of the many patent explosives now in existence* :—

CLASS I. NITRATED POWDERS.

<i>Bolton's Powder.</i>		<i>Pyronome.</i>	
	Parts.		Parts.
Carbonate of copper	8	Nitrate of soda	52.5
Graphite	10	Spent tan	27.5
Prepared quicklime	14	Sulphur	20.0
„ alum	50	<i>Diorrexin (an Austrian explosive).</i>	
„ sugar	350	Nitrates of potash and soda	60
Nitrate of soda	350	Sulphur	12
Soda ash	20	Sawdust	10
Ferrocyanide of potassium . .	300	Charcoal	7
Charcoal	30	Picric acid	1.5
Carbonate of potash	450	Moisture	7.5

* For further information with regard to the preparation, composition, &c., of various explosives, see Major Cundill's "Dictionary of Explosives" and Mr. Eissler's "Handbook of Modern Explosives."

CLASS I. NITRATED POWDERS (*continued*).

<i>Johnite.</i>		<i>Davey's Powder.</i>	
	Parts.		Parts.
Nitrate of potash	75	Saltpetre	64
Sulphur	10	Sulphur	16
Lignite	10	Charcoal	12
Picrate of soda	3	Flour, bran, or starch . .	8
Chlorate of potash	2		
<i>Pudrolithe.</i>		<i>Pyrolithe.</i>	
Saltpetre	68	Saltpetre	51.5
Sulphur	12	Nitrate of soda	16.0
Charcoal	6	Sulphur	20.0
Nitrate of baryta	3	Sawdust	11.0
„ „ soda	3	Charcoal	1.5
Sawdust	5		
Spent tan	3	<i>Safety Blasting Powder.</i>	
		Saltpetre	70
		Sulphur	12
		Lampblack	5
		Spent tan or sawdust . .	13
		Sulphate of iron	2
<i>Courteille's Powder.</i>		<i>Schäffer's Powder.</i>	
Nitrate of soda or potash	60 to 75	Saltpetre	30 to 38
Sulphur	10 „ 12	Nitrate of soda	40
Charcoal	7 „ 10	Sulphur	8 to 12
Peat and hard coal . . .	9 „ 12	Charcoal	7 „ 8
Combined metallic sul-		Rochelle salt	4 „ 6
phates	2 „ 4		
Oleaginous matter,		<i>Violette's Powder.</i>	
animal or vegetable		Nitrate of soda	62.5
(or tar), refined or	1 „ 3	Acetate of soda	37.5
crude			

CLASS II. CHLORATE POWDERS.

<i>Dynamogen.</i>		<i>Jaline.</i>	
	Parts		Parts.
Yellow prussiate of potash .	17	Nitrate of potash	65 to 75
Water	150	Sulphur	10
Charcoal	17	Lignite	10 to 50
After boiling, and cooled,		Picrate of soda	3 „ 8
there are added:—		Chlorate of potash	2
Potash	35		
Chlorate of potash	70	<i>Knaffl's Powder.</i>	
Starch	10	Chlorate of potash	46
Water	50	Nitrate of potash	26
		Sulphur	15
		Ultmate of ammonia . . .	10

CLASS II. CHLORATE POWDERS (*continued*).*Ehrhardt's Powders.*

	Artillery. Pts.	Blasting. Pts.	Shells Pts.
Chlorate of potash	1	1	1
Saltpetre . . .	1	1	—
Charcoal . . .	—	4	—
Tannic acid . .	1	2	1

Goetz' Powder.

	Parts
Chlorate of potash . . .	10
Glucose solution . . .	10
Charcoal in powder . . .	3
Sulphur	2
Amorphous phosphorous . .	1
Picrate of lead	3

Nisger's Powder.

	Parts.
Yellow prussiate of potash .	1'5
Bichromate of potash . . .	2'0
Chlorate of potash	10'5
Nitrates of soda and potash .	44'5
Vegetable matter	6'5
Mineral and vegetable carbon	19'5
Sulphur	15'5

Pyronome.

Saltpetre	69
Sulphur	9
Charcoal	10
Antimony	8
Chlorate of potash	5
Rye flour	4

CLASS III. DYNAMITE POWDERS.

Atlas Powders.

	A. Pts.	B. Pts.
Sodium nitrate	2	34
Wood fibre	21	14
Magnesium carbonate . . .	2	2
Nitroglycerine	75	50

Castellanos Powder.

	Parts
Nitroglycerine	40
Nitrate of Potash	25
Picrate	10
Sulphur	5
Insoluble salts (silicates, &c.)	10
Carbon	10

Petralithe.

Nitroglycerine	640
Nitrate of ammonia, soda or urea	120
Palmitate of cetyl	2½
Carbonate of lime	2½
Animal or vegetable charcoal	230
Bicarbonate of soda	5

Vulcan Powder.

	Parts.
Nitroglycerine	30
Nitrate of soda	52'5
Sulphur	7
Charcoal	10'5

Diaspon.

Nitroglycerine	47 to 63
Nitrocotton	0'5 „ 3'0
Nitrate of soda	22 „ 23
Wood cellulose	8 „ 18
Sulphur	3 „ 9

Dualine.

Nitroglycerine	50
Fine sawdust	30
Saltpetre	20

Hercules Powders.

	No. 1. Pts.	No. 2. Pts.
Carbonate of magnesia	20'85	10'00
Nitrate of potash	2'10	31'00
Chlorate of potash	1'05	3'34
White Sugar	1'00	15'66
Nitroglycerine	75'00	40'00

CLASS III. DYNAMITE POWDERS (*continued*).

<i>Rendrock.</i>		<i>Vigorite.</i>	
	Parts.		Parts.
Nitrate of potash . . .	40	Nitroglycerine . . .	30
Nitroglycerine . . .	40	Nitrate of soda . . .	60
Wood fibre . . .	13	Charcoal . . .	5
Paraffin or pitch . . .	7	Sawdust . . .	5

CLASS IV. GUNCOTTON CLASS.

<i>Ammonia Nitrate Powder.</i>		<i>Johnson's Powders.</i>	
	Parts		For Military Arms. Parts.
Ammonium nitrate . . .	80	Nitrocellulose . . .	50
Potassium chlorate . . .	5	Potassium nitrate . . .	40
Nitroglucose . . .	10	Torrefied starch . . .	10
Coal tar . . .	5		
<i>Nitronaphthalene.</i>		<i>Bellite, Securite, Roburite.</i>	
Nitronaphthalene . . .	10	These explosives, which have advantages for firing in coal mines, are mixtures of nitrate of ammonium with di or tri nitro-benzole, or chlorinated di nitro-benzole.	
Saltpetre . . .	75	<i>Schultze Powder.</i>	
Charcoal . . .	12.5		
Sulphur . . .	12.5		
<i>Petrofacteur.</i>			
Nitro-benzene . . .	10	Soluble nitrolignin . . .	24.83
Chlorate of potash . . .	67	Insoluble nitrolignin . . .	23.36
Nitrate of potash . . .	20	Lignin . . .	13.14
Sulphide of antimony . . .	3	Nitrate of potash and barium . . .	32.35
		Paraffin . . .	3.65
		Matters soluble in alcohol . . .	0.11
		Moisture . . .	2.56

CLASS V. PICRIC POWDERS.

<i>Designolles Powders.</i>			<i>Victorite.</i>	
	For Torpedoes and Shells. Pts.	Small Arms. Pts.		Parts.
Picrate of potash	55	28.6	Chlorate of potash . . .	80
Saltpetre . . .	45	65.0	Picric acid . . .	110
Charcoal . . .	—	6.4	Nitrate of potash . . .	10
			Charcoal . . .	5

Bronolithe.

						Per cent.
Barium sodium picrate	15 to 30
Lead " "	8 " 30
Potassium " "	2 " 10
Nitronaphthalene	5 " 20
Saltpetre	20 " 40
Sugar	1.5 " 3
Gum	2 " 3
Lampblack	0.5 " 4

APPENDIX.

Symbols and Atomic Weights of the Elements.

Element.	Symbol.	Atomic wt.	Observer.
Aluminium	Al	27.02	Mallet.
Antimony	Sb	120.00	Schneider, Cooke.
Arsenic	As	75.15	Kessler.
Barium	Ba	136.84	Marignac.
Bismuth	Bi	210.00	Dumas.
Boron	B	11.04	Berzelius.
Bromine	Br	79.76	Stas.
Cadmium	Cd	112.04	Lenssen.
Cæsium	Cs	133.00	Bunsen.
Calcium	Ca	39.90	Erdmann.
Carbon	C	11.97	Dumas, Liebig.
Cerium	Ce	138.24	Rammelsberg.
Chlorine	Cl	35.37	Stas.
Chromium	Cr	52.08	Siewart.
Cobalt	Co	58.74	Russell.
Copper	Cu	63.12	Millon & Commaille.
Didymium	D	142.44	Hermann.
Erbium	E	168.90	Bahr & Bunsen.
Fluorine	F	18.96	Luca, Louyet.
Gallium	Ga	69.80	Lecoq de Boisbaudran.
Glucinum	Gl	9.30	Awdejew, Klatzo.
Gold	Au	196.71	Berzelius.
Hydrogen	H	1.00	Dulong & Berzelius.
Indium	In	113.40	Winkler, Bunsen.

Element.	Symbol.	Atomic wt.	Observer.
Iodine	I	126·54	Stas.
Iridium	Ir	196·87	Berzelius.
Iron	Fe	56·00	Dumas.
Lanthanum	La	139·33	Hermann.
Lead	Pb	206·40	Stas.
Lithium	Li	7·00	Stas.
Magnesium	Mg	23·94	Dumas.
Manganese	Mn	54·04	Schneider.
Mercury	Hg	200·00	Erdmann.
Molybdenum	Mo	96·00	Dumas, Debray.
Nickel	Ni	58·74	Russell.
Niobium	Nb	94·00	Marignac.
Nitrogen	N	14·01	Stas.
Osmium	Os	199·03	Berzelius.
Oxygen	O	15·96	
Palladium	Pd	106·57	Berzelius.
Phosphorus	P	30·96	Schlotter.
Platinum	Pt	194·38	Seubert.
Potassium	K	39·04	Stas.
Rhodium	Rh	104·21	Berzelius.
Rubidium	Rb	85·40	Bunsen, Piccard.
Ruthenium	Ru	104·40	Berzelius.
Selenium	Se	79·46	Dumas.
Silver	Ag	107·67	Stas.
Silicon	Si	28·10	Dumas.
Sodium	Na	22·99	Stas.
Strontium	Sr	87·54	Marignac.
Sulphur	S	31·996	Stas.
Tantalum	Ta	182·300	Marignac.
Tellurium	Te	128·06	V. Hauer.
Thallium	Tl	203·66	Crookes.
Thorium	Th	231·44	Delafontaine.
Tin	Sn	118·10	Dumas.
Titanium	Ti	50·00	Pierre.
Tungsten	W	184·00	Schneider, Roscoe.
Uranium	U	237·60	Ebelman.
Vanadium	V	51·35	Roscoe.
Yttrium	Y	92·55	Bahr & Bunsen.
Zinc	Zn	65·16	Oxel Erdmann.
Zirconium	Zr	89·60	Marignac.

Table of Atomic Weights.

(Issued December 6, 1890.)

Revised for the Committee of Revision and Publication of the Pharmacopœia of the United States of America. By F. W. Clarke, Chief Chemist of the United States Geological Survey.

This table represents the latest and most trustworthy results, reduced to a uniform basis of comparison, with oxygen = 16 as starting point of the system. No decimal places representing large uncertainties are used. When values vary, with equal probability on both sides, so far as our present knowledge goes, as in the case of a cadmium (111·8 and 112·2), the mean value is given in the Table.

The names of elements occurring in pharmacopœial, medicinal, chemicals, are printed in italics.

Name.	Symhol.	Atomic weight.
<i>Aluminium</i>	<i>Al</i>	27·
<i>Antimony</i>	<i>Sb</i>	120·
<i>Arsenic</i>	<i>As</i>	75·
<i>Barium</i>	<i>Ba</i>	137·
<i>Bismuth</i>	<i>Bi</i>	208·9
<i>Boron</i>	<i>B</i>	11·
<i>Bromine</i>	<i>Br</i>	79·95
<i>Cadmium</i>	<i>Cd</i>	112·
<i>Cæsium</i>	<i>Cs</i>	132·9
<i>Calcium</i>	<i>Ca</i>	40·
<i>Carbon</i>	<i>C</i>	12·
<i>Cerium</i>	<i>Ce</i>	140·2
<i>Chlorine</i>	<i>Cl</i>	35·45
<i>Chromium</i>	<i>Cr</i>	52·1
<i>Cobalt</i>	<i>Co</i>	59·
* <i>Columbium</i>	<i>Cb</i>	94·
<i>Copper</i>	<i>Cu</i>	63·4
† <i>Didymium</i>	<i>Di</i>	142·3
<i>Erbium</i>	<i>Er</i>	166·3
<i>Fluorine</i>	<i>F</i>	19·
<i>Gallium</i>	<i>Ga</i>	69·
<i>Germanium</i>	<i>Ge</i>	72·3
‡ <i>Glucinum</i>	<i>Gl</i>	9·
<i>Gold</i>	<i>Au</i>	197·3
<i>Hydrogen</i>	<i>H</i>	1·007
<i>Indium</i>	<i>In</i>	113·7

* Has priority over niobium.

† Now split into neo- and praseo-didymium.

‡ Has priority of over beryllium.

Name.	Symbol.	Atomic weight.
<i>Iodine</i>	<i>I</i>	126·85
<i>Iridium</i>	<i>Ir</i>	193·1
<i>Iron</i>	<i>Fe</i>	56·
<i>Lanthanum</i>	<i>La</i>	138·2
<i>Lead</i>	<i>Pb</i>	206·95
<i>Lithium</i>	<i>Li</i>	7·02
<i>Magnesium</i>	<i>Mg</i>	24·3
<i>Manganese</i>	<i>Mn</i>	55·
<i>Mercury</i>	<i>Hg</i>	200·
<i>Molybdenum</i>	<i>Mo</i>	96·
<i>Nickel</i>	<i>Ni</i>	58·7
<i>Nitrogen</i>	<i>N</i>	14·03
<i>Osmium</i>	<i>Os</i>	191·7
§ <i>Oxygen</i>	<i>O</i>	16·
<i>Palladium</i>	<i>Pd</i>	106·6
<i>Phosphorus</i>	<i>P</i>	31·
<i>Platinum</i>	<i>Pt</i>	195·
<i>Potassium</i>	<i>K</i>	39·11
<i>Rhodium</i>	<i>Rh</i>	103·5
<i>Rubidium</i>	<i>Rb</i>	85·5
<i>Ruthenium</i>	<i>Ru</i>	101·6
<i>Samarium</i>	<i>Sm</i>	150·
<i>Scandium</i>	<i>Sc</i>	44·
<i>Selenium</i>	<i>Se</i>	79·
<i>Silicon</i>	<i>Si</i>	28·4
<i>Silver</i>	<i>Ag</i>	107·92
<i>Sodium</i>	<i>Na</i>	23·05
<i>Strontium</i>	<i>Sr</i>	87·6
<i>Sulphur</i>	<i>S</i>	32·06
<i>Tantalum</i>	<i>Ta</i>	182·6
<i>Tellurium</i>	<i>Te</i>	125·
<i>Terbium</i>	<i>Tb</i>	159·5
<i>Thallium</i>	<i>Tl</i>	204·18
<i>Thorium</i>	<i>Th</i>	232·6
<i>Tin</i>	<i>Sn</i>	119·
<i>Titanium</i>	<i>Ti</i>	48·
<i>Tungsten</i>	<i>W</i>	184·
<i>Uranium</i>	<i>U</i>	239·6
<i>Vanadium</i>	<i>V</i>	51·4
<i>Ytterbium</i>	<i>Yb</i>	173·
<i>Yttrium</i>	<i>Yt</i>	89·1
<i>Zinc</i>	<i>Zn</i>	65·3
<i>Zirconium</i>	<i>Zr</i>	90·6

‡ Standard, or basis of the system.

**Factors for Ascertaining the Amount of Constituent
Sought from the Form in which it is Weighed.**

Element.	Form.	Sought.	Factor.
Aluminium	Al_2O_3	Al_2	·5340
Ammonium	NH_4Cl	NH_3	·3180
"	$2 \text{NH}_4\text{Cl}_1\text{PtCl}_4$	2NH_3	·0761
Antimony	Sb_2O_3	Sb_2	·8356
"	Sb_2S_3	Sb_2	·7177
"	Sb_2S_3	Sb_2O_3	·8588
"	Sb_2O_4	Sb_2O_3	·9481
Arsenic	As_2O_3	As_2	·7576
"	As_2O_5	As_2	·6522
"	As_2O_5	As_2O_3	·8609
"	As_2S_3	As_2O_3	·8049
"	As_2S_3	As_2O_5	·9350
"	$(\text{MgAmAsO}_4)_2\text{OH}_2$	As_2O_5	·6053
"	"	As_2O_3	·5211
Barium	BaO	Ba	·8954
"	BaSO_4	BaO	·6567
"	BaCO_3	BaO	·7767
"	$\text{BaF}_2, \text{SiF}_4$	BaO	·5484
Bismuth	Bi_2O_3	Bi_2	·8966
Boron	B_2O_3	B_2	·3143
Bromine	AgBr	Br	·4256
Cadmium	CdO	Cd	·8750
Calcium	CaO	Ca	·7143
"	CaSO_4	CaO	·4118
"	CaCO_3	CaO	·5600
Carbon	CO_2	C	·2727
"	CaCO_3	CO_2	·4400
Chlorine	AgCl	Cl	·2472
"	AgCl	HCl	·2542
Chromium	Cr_2O_3	Cr_2	·6862
"	Cr_2O_3	2CrO_3	1·3138
"	PbCrO_4	CrO_3	·3106
Cobalt	Co	CoO	1·2712
"	$\text{Co}_{12}\text{O}_{19}$	Co_{12}	·6999
"	Co_3O_5	Co_3	·6887
"	CoSO_4	CoO	·4839
"	Co_3O_4	Co_3	·7344
"	$\{ \text{Co}_2\text{O}_3, 3 \text{K}_2\text{O}, \}$	2CoO	·1735
"	$\{ 5 \text{N}_2\text{O}_3, 2 \text{H}_2\text{O} \}$		
"	$2 \text{CoSO}_4 + 3 \text{K}_2\text{SO}_4$	2CoO	·1802
"	"	Co_2	·1417

Element.	Form.	Sought.	Factor.
Copper	CuO	Cu	·7985
"	Cu ₂ S	Cu ₂	·7985
Fluorine	CaF ₂	F ₂	·4872
"	SiF ₄	F ₄	·7308
Hydrogen	H ₂ O	H ₂	·1111
Iodine	AgI	I	·5405
"	PdI ₂	I ₂	·7056
Iron	Fe ₂ O ₃	Fe ₂	·7000
"	Fe ₂ O ₃	2 FeO	·9000
"	FeS	Fe	·6364
Lead	PbO	Pb	·9283
"	PbSO ₄	PbO	·7360
"	PbSO ₄	Pb	·6832
"	PbCl ₂	Pb	·7448
"	PbCl ₂	PbO	·8024
"	PbS	PbO	·9331
Lithium	Li ₂ CO ₃	Li ₂ O	·4054
"	Li ₂ SO ₄	Li ₂ O	·2727
"	Li ₃ PO ₄	Li ₂ O	·3879
Magnesium	MgO	Mg	·6000
"	MgSO ₄	MgO	·3335
"	Mg ₂ P ₂ O ₇	2 MgO	·3604
Manganese	MnO	Mn	·7747
"	Mn ₃ O ₄	Mn ₃	·7205
"	Mn ₂ O ₃	Mn ₂	·6962
"	MnSO ₄	MnO	·4702
"	MnS	MnO	·8161
"	MnS	Mn	·6322
Mercury	2 Hg	Hg ₂ O	1·0400
"	Hg	HgO	1·0800
"	Hg ₂ Cl ₂	2 Hg	·8494
"	HgS	Hg	·8621
"	NiO	Ni	·7867
Nickel		N ₂	·0627
Nitrogen	2 AmCl, PtCl ₄	N ₂	·1416
"	Pt	N ₂	·1416
"	BaSO ₄	N ₂ O ₅	·4635
"	AgCN	CN	·1941
"	AgCN	HCN	·2016
"	Al ₂ O ₃	O ₃	·4660
Oxygen	Sb ₂ O ₃	O ₃	·1644
"	As ₂ O ₃	O ₃	·2424
"	As ₂ O ₅	O ₅	·3478
"	BaO	O	·1046
"	Bi ₂ O ₃	O ₃	·1035

Element.	Form.	Sought.	Factor.
Oxygen	CdO	O	·1250
"	Cr ₂ O ₃	O ₃	·3138
"	CoO	O	·2133
"	CuO	O	·2015
"	FeO	O	·2222
"	Fe ₂ O ₃	O ₃	·3000
"	PbO	O	·0718
"	CaO	O	·2857
"	MgO	O	·3997
"	MnO	O	·2254
"	Mn ₃ O ₄	O ₄	·2795
"	Mn ₂ O ₃	O ₃	·3038
"	Hg ₂ O	O	·0385
"	HgO	O	·0741
"	NiO	O	·2133
"	K ₂ O	O	·1698
"	SiO ₂	O ₂	·5333
"	Ag ₂ O	O	·0690
"	Na ₂ O	O	·2581
"	SrO	O	·1546
"	SnO ₂	O	·2133
"	H ₂ O	O	·8889
"	ZnO	O	·1974
Phosphorus	P ₂ O ₅	P ₂	·4366
"	Mg ₂ P ₂ O ₇	P ₂ O ₅	·6396
"	MgP ₂ O ₇	2 PO ₄	·8559
"	Fe ₂ P ₂ O ₆	P ₂ O ₅	·4702
"	P ₂ O ₅	2 PO ₄	1·3380
"	Ag ₃ PO ₄	(P ₂ O ₅) $\frac{1}{2}$	·1695
"	U ₄ P ₂ O ₁₁	P ₂ O ₅	·1991
"	Ag ₄ P ₂ O ₇	P ₂ O ₅	·2344
Potassium	K ₂ O	K ₂	·8302
"	K ₂ SO ₄	K ₂ O	·5400
"	KNO ₃	(K ₂ O) $\frac{1}{2}$	·4659
Potassium	KCl	K	·5245
"	KCl	(K ₂ O) $\frac{1}{2}$	·6317
"	2 KCl, PtCl ₄	K ₂ O	·1927
"	"	2 KCl	·3070
Silicon	SiO ₂	Si	·4667
Silver	AgCl	Ag	·7528
"	AgCl	(Ag ₂ O) $\frac{1}{2}$	·8085
Sodium	Na ₂ O	Na ₂	·7419
"	Na ₂ SO ₄	Na ₂ O	·4366
"	NaNO ₃	(Na ₂ O) $\frac{1}{2}$	·3647

Element.	Form.	Sought.	Factor.
Sodium	NaCl	$(\text{Na}_2\text{O})\frac{1}{2}$	·5302
"	NaCl	Na	·3934
"	Na_2CO_3	Na_2O	·5849
Strontium	SrO	Sr	·8454
"	SrSO_4	SrO	·5640
"	SrCO_3	SrO	·7017
Sulphur	BaSO_4	S	·1373
"	As_2S_3	S_3	·3902
"	BaSO_4	SO_3	·3434
"	SO_3	SO_4	1·2000
Tin	SnO_2	Sn	·7867
"	SnO_2	SnO	·8933
Zinc	ZnO	Zn	·8026
"	ZnS	ZnO	·8352
"	ZnS	Zn	·6703

English Weights and Measures.

AVOIRDUPOIS.

	Grains.	Drachms.	Ounces.	Lbs.	Qrs.	Cwts.	Tons.
Grain .	1						
Drachm .	27·34	1					
Ounce .	437·5	16	1				
Pound .	7,000·	256	16	1			
Quarter	196,000·	7,168	448	28·	1		
Cwt. .	784,000·	28,672	1,792	112	4	1	
Ton .	15,680,000·	573,440	35,840	2,240	80	20	1

TROY WEIGHT.

	Grains.	Dwts.	Ounces.	Lb.
Grain .	1			
Pennyweight .	24	1		
Ounce .	480	20	1	
Pound .	5,760	240	12	1

	Cubic inches.
1 gallon	= 277.276
1 pint	= 34.659
1 fluid ounce	= 1.7329
1 litre	= 61.02705
1 cubic centimetre	= 0.06102705
1 cubic inch	= 16.386176 cubic centimetres.

1 cubic inch of distilled water in air at 62° F = 252.330 grains.

1 cubic inch „ „ *in vacuo* at 62° F = 252.645 grains.

1 minim is the volume of 0.91 grain of water.

1 fluid drachm „ of 54.68 grains of water.

1 fluid ounce „ of 437.5 grains of water.

1 gallon is the „ of 10 pounds or 70,000 grains of water.

Weights and Measures of the British Pharmacopœia of 1867.

WEIGHTS.

1 grain, gr.

1 ounce, oz. = 437.5 grains.

1 pound = 16 ozs. = 7,000 „

MEASURES OF CAPACITY.

1 minim, min.

1 fluid drachm, fl. dr. = 60 minims.

1 fluid ounce, fl. oz. = 8 fluid drachms.

1 pint, O = 20 fluid ounces.

1 gallon, C = 8 pints.

MEASURES OF LENGTH.

1 line = $\frac{1}{12}$ inch.

1 inch = $\frac{1}{39.1393}$ seconds—pendulum.

12 „ = 1 foot.

36 „ = 3 feet = 1 yard.

Weights and Measures of the Metrical System.

WEIGHTS.

1 milligram	=	·001	gram.
1 centigram	=	·01	„
1 decigram	=	0·1	„
1 gram.	=	weight of a cubic centimetre of water at 4° C.	
1 decagram	=	10	grms.
1 hectogram	=	100	„
1 kilogram	=	1,000	„

MEASURES OF CAPACITY.

1 millilitre	=	1	cubic centimetre of water at 4° C.
1 centilitre	=	10	cubic centimetres:
1 decilitre	=	100	„ „
1 litre	=	1,000	„ „

MEASURES OF LENGTH.

1 millimetre	=	·001	metre.
1 centimetre	=	·01	„
1 decimetre	=	0·1	„
1 metre	=	the ten millionth part of a quarter of the earth's meridian (nearly).	

Tables for Conversion of Metrical and English Measures.

A. LENGTH.

METRICAL TO ENGLISH.

ENGLISH TO METRICAL.

(1) Millimetres to inches.	(2) Metres to feet.	(3) Inches to millimetres.	(4) Feet to metres.
1 = 0·03937079	1 = 3·2808992	1 = 25·39954	1 = 0·30479449
2 = 0·07874158	2 = 6·5617984	2 = 50·79908	2 = 0·60958898
3 = 0·11811237	3 = 9·8426976	3 = 76·19862	3 = 0·91438347
4 = 0·15748316	4 = 13·1235968	4 = 101·59816	4 = 1·21917796
5 = 0·19685395	5 = 16·4044960	5 = 126·99770	5 = 1·52397245
6 = 0·23622474	6 = 19·6853952	6 = 152·39724	6 = 1·82876694
7 = 0·27550553	7 = 22·9662944	7 = 177·79678	7 = 2·13356143
8 = 0·31496632	8 = 26·2471936	8 = 203·19632	8 = 2·43835592
9 = 0·35433711	9 = 29·5280928	9 = 228·59586	9 = 2·74315041

B. CAPACITY.

METRICAL TO ENGLISH.

(1) Cubic centimetres to cubic inches.	(2) Litres to fluid ounces.	(3) Litres to pints.	(4) Litres to gallons.
1 = 0.06102705	1 = 35.215468	1 = 1.7607734	1 = 0.22009668
2 = 0.12205410	2 = 70.430936	2 = 3.5215468	2 = 0.44019336
3 = 0.18308115	3 = 105.646404	3 = 5.2823202	3 = 0.66029004
4 = 0.24410820	4 = 140.861872	4 = 7.0430936	4 = 0.88038672
5 = 0.30513525	5 = 176.077340	5 = 8.8038670	5 = 1.10048340
6 = 0.36616230	6 = 211.292808	6 = 10.5646404	6 = 1.32058008
7 = 0.42718935	7 = 246.508276	7 = 12.3254138	7 = 1.54067676
8 = 0.48821640	8 = 281.723744	8 = 14.0861872	8 = 1.76077344
9 = 0.54924345	9 = 316.939212	9 = 15.8469606	9 = 1.98087012

ENGLISH TO METRICAL.

(1) Cubic inches to cubic centimetres.	(2) Fluid ounces to cubic centimetres.	(3) Pints to litres.	(4) Gallons to litres.
1 = 16.386176	1 = 28.396612	1 = 0.567932	1 = 4.543458
2 = 32.772352	2 = 56.793224	2 = 1.135864	2 = 9.086916
3 = 49.158528	3 = 85.189856	3 = 1.703796	3 = 13.630374
4 = 65.544704	4 = 113.586448	4 = 2.271728	4 = 18.173832
5 = 81.930880	5 = 141.983060	5 = 2.839660	5 = 22.717290
6 = 98.317056	6 = 170.379672	6 = 3.407592	6 = 27.270748
7 = 114.703232	7 = 198.776284	7 = 3.975524	7 = 31.804206
8 = 131.089408	8 = 227.172896	8 = 4.543456	8 = 36.347664
9 = 147.475584	9 = 255.569508	9 = 5.111388	9 = 40.891122

C. WEIGHT.

METRICAL TO ENGLISH.

(1) Grammes to grains.	(2) Kilogrammes to ounces.	(3) Kilogrammes to pounds.
1 = 15.4323488	1 = 35.27394	1 = 2.20462
2 = 30.8646976	2 = 70.54788	2 = 4.40924
3 = 46.2970464	3 = 105.82182	3 = 6.61386
4 = 61.7293952	4 = 141.09576	4 = 8.81848
5 = 77.1617440	5 = 176.36970	5 = 11.02310
6 = 92.5940928	6 = 211.64364	6 = 13.22772
7 = 108.0264416	7 = 246.91758	7 = 15.43234
8 = 123.4587904	8 = 282.19152	8 = 17.63696
9 = 138.8911392	9 = 317.46546	9 = 19.84158

ENGLISH TO METRICAL.

(1) Grains to grammes.	(2) Ounces to grammes.	(3) Pounds to kilogrammes.	(4) Hundredweights to kilogrammes.
1 = 0.0647989	1 = 28.34954	1 = 0.45359265	1 = 50.8023768
2 = 0.1295978	2 = 56.69908	2 = 0.90718530	2 = 101.6047536
3 = 0.1943967	3 = 85.04862	3 = 1.36077795	3 = 152.4071304
4 = 0.2591956	4 = 113.39816	4 = 1.81437060	4 = 203.2095072
5 = 0.3239945	5 = 141.74770	5 = 2.26796325	5 = 254.0118840
6 = 0.3887934	6 = 170.09724	6 = 2.72155590	6 = 304.8142608
7 = 0.4535923	7 = 198.44678	7 = 3.17514855	7 = 355.6166376
8 = 0.5183912	8 = 226.79632	8 = 3.62874120	8 = 406.4190144
9 = 0.5831901	9 = 255.14586	9 = 4.08233385	9 = 457.2213912

Hydrometer Tables.

DENSITIES CORRESPONDING TO DEGREES OF BAUMÉ'S HYDROMETER
FOR LIQUIDS HEAVIER THAN WATER.

° B.	Density.	° B.	Density.	° B.	Density.	° B.	Density.
0	1.000	19	1.152	38	1.357	57	1.652
1	1.007	20	1.161	39	1.370	58	1.671
2	1.014	21	1.171	40	1.383	59	1.691
3	1.022	22	1.180	41	1.397	60	1.711
4	1.029	23	1.190	42	1.410	61	1.732
5	1.036	24	1.199	43	1.424	62	1.753
6	1.044	25	1.210	44	1.438	63	1.774
7	1.052	26	1.221	45	1.453	64	1.796
8	1.060	27	1.231	46	1.468	65	1.819
9	1.067	28	1.242	47	1.483	66	1.846
10	1.075	29	1.253	48	1.498	67	1.872
11	1.083	30	1.264	49	1.514	68	1.897
12	1.091	31	1.275	50	1.530	69	1.921
13	1.100	32	1.286	51	1.546	70	1.946
14	1.108	33	1.297	52	1.563	71	1.974
15	1.116	34	1.309	53	1.580	72	2.000
16	1.125	35	1.320	54	1.597	73	2.031
17	1.134	36	1.332	55	1.615	74	2.059
18	1.143	37	1.345	56	1.634		

**DENSITIES CORRESPONDING TO BAUMÉ'S HYDROMETER FOR LIQUIDS
LIGHTER THAN WATER. (FRANCŒUR).**

° B.	Density.	° B.	Density.	° B.	Density.	° B.	Density.
10	1·000	23	0·918	36	0·849	49	0·789
11	0·993	24	0·913	37	0·844	50	0·785
12	0·986	25	0·907	38	0·839	51	0·781
13	0·980	26	0·901	39	0·834	52	0·777
14	0·973	27	0·896	40	0·830	53	0·773
15	0·967	28	0·890	41	0·825	54	0·768
16	0·960	29	0·885	42	0·820	55	0·764
17	0·954	30	0·880	43	0·816	56	0·760
18	0·948	31	0·874	44	0·811	57	0·757
19	0·942	32	0·869	45	0·807	58	0·753
20	0·936	33	0·864	46	0·802	59	0·749
21	0·930	34	0·859	47	0·798	60	0·745
22	0·924	35	0·854	48	0·794		

**DEGREES ON TWADDLE'S HYDROMETER WITH THE CORRESPONDING
DENSITIES.**

° Tw.	Density.	° Tw.	Density.	° Tw.	Density.	° Tw.	Density.
1	1·005	8	1·040	15	1·075	22	1·110
2	1·010	9	1·045	16	1·080	23	1·115
3	1·015	10	1·050	17	1·085	24	1·120
4	1·020	11	1·055	18	1·090	25	1·125
5	1·025	12	1·060	19	1·095	26	1·130
6	1·030	13	1·065	20	1·100	27	1·135
7	1·035	14	1·070	21	1·105	28	1·140

Degrees Twaddle are converted into the corresponding specific gravities by multiplying them by 5 and adding 1,000.

Comparison of Thermometers.

CONVERSION OF THERMOMETER DEGREES.

° C. to ° R. : multiply by 4 and divide by 5.

° C. to ° F. : multiply by 9, divide by 5, then add 32.

° R. to ° C. : multiply by 5 and divide by 4.

° R. to ° F. : multiply by 9, divide by 4, then add 32.

° F. to ° R. : first subtract 32, then multiply by 4, and divide by 9.

° F. to ° C. : first subtract 32, then multiply by 5 and divide by 9.

Comparison of Centigrade and Fahrenheit Degrees.

° C.	° F.	° C.	° F.	° C.	° F.	° C.	° F.
+ 500	+ 932	+ 74	+ 165.2	+ 45	+ 113	+ 16	+ 60.8
400	752	73	163.4	44	111.2	15	59
300	572	72	161.6	43	109.4	14	57.2
200	392	71	159.8	42	107.6	13	55.4
100	212	70	158	41	105.8	12	53.6
99	210.2	69	156.2	40	104	11	51.8
98	208.4	68	154.4	39	102.2	10	50
97	206.6	67	152.6	38	100.4	9	48.2
96	204.8	66	150.8	37	98.6	8	46.4
95	203	65	149	36	96.8	7	44.6
94	201.2	64	147.2	35	95	6	42.8
93	199.4	63	145.4	34	93.2	5	41
92	197.6	62	143.6	33	91.4	4	39.2
91	195.8	61	141.8	32	89.6	3	37.4
90	194	60	140	31	87.8	2	35.6
89	192.2	59	138.2	30	86	1	33.8
88	190.4	58	136.4	29	84.2	0	32
87	188.6	57	134.6	28	82.4	— 1	30.2
86	186.8	56	132.8	27	80.6	2	28.4
85	185	55	131	26	78.8	3	26.6
84	183.2	54	129.2	25	77	4	24.8
83	181.4	53	127.4	24	75.2	5	23
82	179.6	52	125.6	23	73.4	6	21.2
81	177.8	51	123.8	22	71.6	7	19.4
80	176	50	122	21	69.8	8	17.6
79	174.2	49	120.2	20	68	9	15.8
78	172.4	48	118.4	19	66.2	10	14
77	170.6	47	116.6	18	64.4	11	12.2
76	168.8	46	114.8	17	62.6	12	10.4
75	167						

Percentages and Gravity of Sulphuric Acid.

TABLE SHOWING PERCENTAGES OF REAL SULPHURIC ACID (H_2SO_4)
CORRESPONDING TO VARIOUS SPECIFIC GRAVITIES OF AQUEOUS
SULPHURIC ACID.

Bineau ; Otto. Temp. 15° C.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1·8426	100	1·675	75	1·398	50	1·182	25
1·842	99	1·663	74	1·3886	49	1·174	24
1·8406	98	1·651	73	1·379	48	1·167	23
1·840	97	1·639	72	1·370	47	1·159	22
1·8384	96	1·627	71	1·361	46	1·1516	21
1·8376	95	1·615	70	1·351	45	1·144	20
1·8356	94	1·604	69	1·342	44	1·136	19
1·834	93	1·592	68	1·333	43	1·129	18
1·831	92	1·580	67	1·324	42	1·121	17
1·827	91	1·568	66	1·315	41	1·1136	16
1·822	90	1·557	65	1·306	40	1·106	15
1·816	89	1·545	64	1·2976	39	1·098	14
1·809	88	1·534	63	1·289	38	1·091	13
1·802	87	1·523	62	1·281	37	1·083	12
1·794	86	1·512	61	1·272	36	1·0756	11
1·786	85	1·501	60	1·264	35	1·068	10
1·777	84	1·490	59	1·256	34	1·061	9
1·767	83	1·480	58	1·2476	33	1·0536	8
1·756	82	1·469	57	1·239	32	1·0464	7
1·745	81	1·4586	56	1·231	31	1·039	6
1·734	80	1·448	55	1·223	30	1·032	5
1·722	79	1·438	54	1·215	29	1·0256	4
1·710	78	1·428	53	1·2066	28	1·019	3
1·698	77	1·418	52	1·198	27	1·013	2
1·686	76	1·408	51	1·190	26	1·0064	1

Percentages and Gravity of Hydrochloric Acid.

TABLE GIVING THE PERCENTAGES OF HYDROCHLORIC ACID CONTAINED
IN AQUEOUS SOLUTIONS OF THE GAS OF VARIOUS SPECIFIC
GRAVITIES.

Ure. Temp. 15° C.

Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.
1.200	40.777	1.1515	30.582	1.1000	20.388	1.0497	10.194
1.1982	40.369	1.1494	30.174	1.0980	19.980	1.0477	9.786
1.1964	39.961	1.1473	29.767	1.0960	19.572	1.0457	9.379
1.1946	39.554	1.1452	29.359	1.0939	19.165	1.0437	8.971
1.1928	39.146	1.1431	28.951	1.0919	18.757	1.0417	8.563
1.1910	38.738	1.141	28.544	1.0899	18.349	1.0397	8.155
1.1893	38.330	1.1389	28.136	1.0879	17.941	1.0377	7.747
1.1875	37.923	1.1369	27.728	1.0859	17.534	1.0357	7.340
1.1857	37.516	1.1349	27.321	1.0838	17.126	1.0337	6.932
1.1846	37.108	1.1328	26.913	1.0818	16.718	1.0318	6.524
1.1822	36.700	1.1308	26.505	1.0798	16.310	1.0298	6.116
1.1802	36.292	1.1287	26.098	1.0778	15.902	1.0279	5.709
1.1782	35.884	1.1267	25.690	1.0758	15.494	1.0259	5.301
1.1762	35.476	1.1247	25.282	1.0738	15.087	1.0239	4.893
1.1741	35.068	1.1226	24.874	1.0718	14.679	1.0220	4.486
1.1721	34.660	1.1206	24.466	1.0697	14.271	1.0200	4.078
1.1701	34.252	1.1185	24.058	1.0677	13.863	1.0180	3.670
1.1681	33.845	1.1164	23.650	1.0657	13.456	1.0160	3.262
1.1661	33.437	1.1143	23.242	1.0637	13.049	1.0140	2.854
1.1641	33.029	1.1123	22.834	1.0617	12.641	1.0120	2.447
1.1620	32.621	1.1102	22.426	1.0597	12.233	1.0100	2.039
1.1599	32.213	1.1082	22.019	1.0577	11.825	1.0080	1.631
1.1578	31.805	1.1061	21.611	1.0557	11.418	1.0060	1.224
1.1557	31.398	1.1041	21.203	1.0537	11.010	1.0040	.816
1.1536	30.990	1.1020	20.796	1.0517	10.602	1.0020	.408

Percentages and Gravity of Nitric Acid.

TABLE SHOWING THE PERCENTAGES OF NITRIC ACID (HNO_3) IN
AQUEOUS SOLUTIONS OF VARIOUS SPECIFIC GRAVITIES.

Kolb, Ann. Ch. Phys. (4) 136. Temp. 15°C .

HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.
100.00	1.530	80.96	1.463	59.59	1.372	39.00	1.244
99.84	1.530	80.00	1.460	58.88	1.368	37.95	1.237
99.72	1.530	79.00	1.456	58.00	1.363	36.00	1.225
99.52	1.529	77.66	1.451	57.00	1.358	35.00	1.218
97.89	1.523	76.00	1.445	56.10	1.353	33.86	1.211
97.00	1.520	75.00	1.442	55.00	1.346	32.00	1.198
96.00	1.516	74.01	1.438	54.00	1.341	31.00	1.192
95.27	1.514	73.00	1.435	53.81	1.339	30.00	1.185
94.00	1.509	72.39	1.432	53.00	1.335	29.00	1.179
93.01	1.506	71.24	1.429	52.33	1.331	28.00	1.172
92.00	1.503	69.96	1.423	50.99	1.323	27.00	1.166
91.00	1.499	69.20	1.419	49.97	1.317	25.71	1.157
90.00	1.495	68.00	1.414	49.00	1.312	23.00	1.138
89.56	1.494	67.00	1.410	48.00	1.304	20.00	1.120
88.00	1.488	66.00	1.405	47.18	1.298	17.47	1.105
87.45	1.486	65.07	1.400	46.64	1.295	15.00	1.089
86.17	1.482	64.00	1.395	45.00	1.284	13.00	1.077
85.00	1.478	63.59	1.393	43.53	1.274	11.41	1.067
84.00	1.474	62.00	1.386	42.00	1.264	7.22	1.045
83.00	1.470	61.21	1.381	41.00	1.257	4.00	1.022
82.00	1.467	60.00	1.374	40.00	1.251	2.00	1.010

Oxide of Potassium and Caustic Potash.

TABLE SHOWING THE PERCENTAGE OF OXIDE OF POTASSIUM (K_2O) IN AQUEOUS SOLUTIONS OF CAUSTIC POTASH (KHO) OF VARIOUS SPECIFIC GRAVITIES.Tünnermann. Temp. $15^{\circ}C$.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1.3300	28.290	1.1437	14.145
1.3131	27.158	1.1308	13.013
1.2966	26.027	1.1182	11.882
1.2805	24.895	1.1059	10.750
1.2648	23.764	1.0938	9.619
1.2493	22.632	1.0819	8.487
1.2342	21.500	1.0703	7.355
1.2268	20.935	1.0589	6.224
1.2122	19.803	1.0478	5.002
1.1979	18.671	1.0369	3.961
1.1839	17.540	1.0260	2.829
1.1702	16.408	1.0153	1.697
1.1568	15.277	1.0050	.5658

Oxide of Sodium and Caustic Soda.

TABLE SHOWING THE PERCENTAGE OF OXIDE OF SODIUM (Na_2O) IN AQUEOUS SOLUTIONS OF CAUSTIC SODA ($NaHO$) OF VARIOUS SPECIFIC GRAVITIES.

Tünnermann.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1.4285	30.220	1.3198	22.363	1.2392	15.110	1.1042	7.253
1.4193	29.616	1.3143	21.894	1.2280	14.500	1.0948	6.648
1.4101	29.011	1.3125	21.758	1.2178	13.901	1.0855	6.044
1.4011	28.407	1.3053	21.154	1.2058	13.297	1.0764	5.440
1.3923	27.802	1.2982	20.550	1.1948	12.692	1.0675	4.835
1.3836	27.200	1.2912	19.945	1.1841	12.088	1.0587	4.231
1.3751	26.594	1.2843	19.341	1.1734	11.484	1.0500	3.626
1.3668	25.989	1.2775	18.730	1.1630	10.879	1.0414	3.022
1.3586	25.385	1.2708	18.132	1.1528	10.275	1.0330	2.418
1.3505	24.780	1.2642	17.528	1.1428	9.670	1.0246	1.813
1.3426	24.176	1.2578	16.923	1.1330	9.066	1.0163	1.209
1.3349	23.572	1.2515	16.379	1.1233	8.462	1.0081	0.604
1.3273	22.967	1.2453	15.714	1.1137	7.857	1.0040	0.302

Percentages and Gravity of Ammonia.

TABLE SHOWING THE PERCENTAGES OF AMMONIA (NH_3) IN AQUEOUS SOLUTIONS OF THE GAS OF VARIOUS SPECIFIC GRAVITIES.Carius. Temp. 15°C .

Specific gravity.	NH_3 Per cent.	Specific gravity.	NH_3 Per cent.	Specific gravity.	NH_3 Per cent.
0.8844	36	0.9133	24	0.9520	12
0.8864	35	0.9162	23	0.9556	11
0.8885	34	0.9191	22	0.9593	10
0.8907	33	0.9221	21	0.9631	9
0.8929	32	0.9251	20	0.9670	8
0.8953	31	0.9283	19	0.9709	7
0.8976	30	0.9314	18	0.9749	6
0.9001	29	0.9347	17	0.9790	5
0.9026	28	0.9380	16	0.9831	4
0.9052	27	0.9414	15	0.9873	3
0.9078	26	0.9449	14	0.9915	2
0.9106	25	0.9484	13	0.9959	1

Melting Points of the Metals.

	Melting point $^\circ \text{C}$.		Melting point $^\circ \text{C}$.
Aluminium	700	Lithium	180
Antimony	425	Magnesium	232
Arsenic	415	Mercury	-40
Bismuth	270	Nickel	1,500—1,600
Cadmium	320	Phosphorus	44
Cobalt	1,050—1,200	Potassium	62.5
Copper	1,050	Platinum	2,600
Gold	1,250	Silver	1,000
Indium	176	Selenium	217
Iron, Cast	1,050—1,200	Sodium	96
„ Steel	1,300—1,400	Sulphur	115
„ Wrought	1,500—1,600	Thallium	290
Lead	330	Tin	235
		Zinc	412

Comparative Wear of Axle-bearings.

(Thurston).

Bearing.	Composition.			Cost per 100 lbs.*	Miles run per lb.	Wear per 100 miles for four bearings.
	Copper.	Tin.	Anti- mony.			
Gun metal . . .	83	17	—	\$ c. 26 60	25,489	grs. 200
" " " " . . .	82	18	—	28 68	27,918	252
White "metal . . .	3	90	7	32 85	22,075	366
" " " " . . .	5	85	10	32 27	24,857	284
Lead composition : lead 84; antimony 16	13 04	22,921	308
Gun metal on brake cars	82	18.	..	28 68	2,576	274

* Including melting expenses, loss, &c. These figures are constantly varying.

Text of the Petroleum Act, 1879.

(42 & 43 VICT. C. 47.)

1. This Act may be cited as the Petroleum Act, 1879.

This Act shall be construed as one with the Petroleum Act, 1871, and together with that Act may be cited as the Petroleum Acts, 1871 and 1879.

2. Whereas by the Petroleum Act, 1871, it is enacted that the term "petroleum to which this Act applies" means such of the petroleum defined by that Act as, when tested in manner set forth in Schedule One to that Act, gives off an inflammable vapour at a temperature of less than one hundred degrees of Fahrenheit's thermometer, and it is expedient to alter the said test: Be it therefore enacted that—

In the Petroleum Act, 1871, the term "petroleum to which this Act applies" shall mean such of the petroleum defined by section three of that Act as, when tested in manner set forth in Schedule One to this Act, gives off an inflammable vapour at a temperature of less than seventy-three degrees of Fahrenheit's thermometer.

Every reference in the Petroleum Act, 1871, to Schedule One to that Act shall be construed to refer to Schedule One to this Act.

3. A model of the apparatus for testing petroleum, as described in Schedule One to this Act, shall be deposited with the Board of Trade, and the Board of Trade shall, on payment of such fee, not exceeding five shillings, as they from time to time prescribe, cause to be compared with such model and verified every apparatus constructed in accordance with Schedule One to this Act which is submitted to them for the purpose, and if the same is found correct shall stamp the same with a mark approved of by the Board and notified in the London Gazette.

An apparatus for testing petroleum purporting to be stamped with the said mark shall, until the contrary is proved, be deemed to have been verified by the Board of Trade.

All fees under this section shall be paid into the Exchequer.

4. The Petroleum Act, 1871, shall continue in force until otherwise directed by Parliament.

5. This Act shall come into operation on the thirty-first day of December one thousand eight hundred and seventy-nine, which day is in this Act referred to as the commencement of this Act.

6. The Petroleum Act, 1871, shall be repealed after the commencement of this Act to the extent in the third column of the Second Schedule to this Act mentioned.

Provided that any sample of petroleum taken before the commencement of this Act shall be tested in manner set forth in Schedule One to the Petroleum Act, 1871, and any offence committed before the commencement of this Act shall

be prosecuted, and any investigation, legal proceeding, or remedy in relation to such offence, or to any act done before the commencement of this Act, shall be instituted, carried on, and have effect as if the provisions of this Act, other than those continuing the Petroleum Act, 1871, had not been passed.

FIRST SCHEDULE.—MODE OF TESTING PETROLEUM SO AS TO ASCERTAIN THE TEMPERATURE AT WHICH IT WILL GIVE OFF INFLAMMABLE VAPOUR. SPECIFICATION OF THE TEST APPARATUS. (See *ante*, p. 204.) DIRECTIONS FOR APPLYING THE FLASHING TEST. (See *ante*, p. 207.)

SECOND SCHEDULE.—ACT REPEALED.

Year and Chapter.	Title.	Extent of repeal.
34 & 35 Vict. c. 105.	The Petroleum Act, 1871.	Section three, from "and the term petroleum to which this Act applies" inclusive to the end of the section. Section eighteen.

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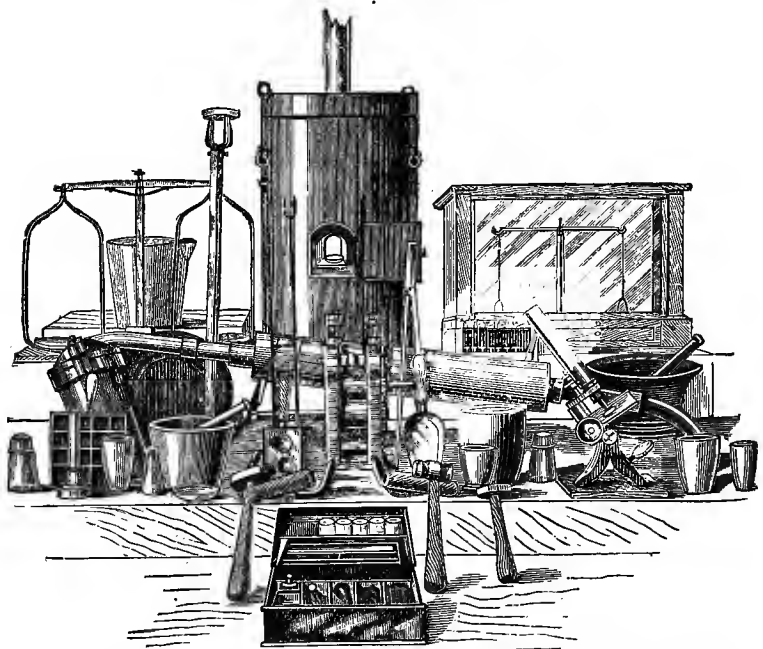
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